

# **ENTROPY**

ENVIRONMENTALISTS INC.

---

POST OFFICE BOX 12291  
RESEARCH TRIANGLE PARK  
NORTH CAROLINA 27709-2291  
919-781-3550

**PROPOSAL TO PERFORM**

**LIQUID WASTE INCINERATOR  
TRIAL BURN SAMPLING AND ANALYSIS**

**FOR**

**ELI LILLY AND COMPANY  
MAYAGUEZ, PUERTO RICO**

**SUBMITTED BY:  
ENTROPY ENVIRONMENTALISTS, INC.**

**JUNE 25, 1986**

PROPOSAL FOR TRIAL BURN SAMPLING AND ANALYSIS  
LIQUID WASTE INCINERATOR

INTRODUCTION

Eli Lilly and Company of Mayaguez, Puerto Rico intends to employ a qualified contractor to assist in conducting trial burn testing of its liquid waste incinerator. Entropy is pleased to offer its services to Eli Lilly and Company in conducting this program.

Entropy's experience in sampling for volatile organics and other hazardous constituents began prior to the passage of RCRA, and prior to the development of many of the test methods currently used for determining compliance with RCRA. Entropy's hazardous emissions testing experience, therefore, reflects our participation in the ongoing evolution of these test methods, a process which still continues. Recent test programs conducted by Entropy incorporate the latest methods and equipment, such as Modified Method 5 (MM5) and VOST. Entropy has performed hazardous waste incinerator tests for Eli Lilly and Company at the Clinton and Lafayette, Indiana facilities.

Entropy will perform sampling and analysis to measure all exhaust stack emissions parameters of interest. Entropy will also receive custody of composite samples of waste feed and scrubber makeup and discharge collected by Eli Lilly and Company personnel in conjunction with the stack testing.

Our proposal discusses the scope of work proposed by Eli Lilly, the sampling equipment and procedures we propose to use and quality assurance considerations.

**ENTROPY**

SECTION 1.  
SCOPE OF WORK

This section describes the trial burn condition and associated sampling planned for the incinerator. More details concerning the sampling and analytical procedures Entropy proposes to follow are presented in Section 2.

1.1 REQUIREMENTS OF A TRIAL BURN.

The incineration standards promulgated by the EPA on January 23, 1981, and amended on June 24, 1982, specify three major requirements regarding hazardous waste incinerator performance. They are:

- 1) that the unit must achieve a destruction and removal efficiency (DRE) of 99.99 percent for all principal organic hazardous constituents (POHC's) designated in the waste feed;
- 2) that emissions of particulate matter must not exceed 0.08 grains per dry standard cubic foot of stack gas, corrected to seven percent oxygen; and,
- 3) that gaseous hydrogen chloride emissions must be limited to four pounds per hour or reduced at a removal efficiency of 99 percent.

These parameters must be determined for each trial burn condition. A condition is defined as a single combination of incinerator operating conditions, waste composition, and waste feed rate.

Entropy will perform sampling and analysis to measure all exhaust stack emissions parameters of interest for one condition. Entropy will also receive custody of composite samples of waste feed and scrubber makeup and discharge collected by plant personnel in conjunction with the stack testing.

## 1.2 TRIAL BURN WASTE COMPOSITION.

A trial burn will be conducted while the incinerator burns primary and secondary wastes at rates of approximately 2.5 and 6.0 gallons per minute, respectively. The principal organic hazardous constituents (POHCs) in the primary waste feed will consist of methylene chloride and carbon tetrachloride. The secondary waste feed will consist of water. The composition and feed rates of the organic constituents in the wastes are shown in Table 1-1.

TABLE 1-1

### TRIAL BURN WASTE ORGANIC CONSTITUENTS

<u>Waste Feed</u>	<u>Constituent</u>	<u>lbs/gallon</u>	<u>lbs/hr</u>
Primary	Methylene Chloride	2.235	335.25
	Carbon Tetrachloride	0.536	80.4

## 1.3 TEST CONDITIONS.

One trial burn test condition is to be performed on the liquid waste incinerator. Incinerator operating parameters are as follows:

Combustion Zone Temperature: 1750-1800°F

Primary Waste Feed Rate: 2-2.5 gpm

Secondary Waste Feed Rate: 5-6 gpm

Combustion Air Feed Rate: 5000-6000 CFM



## SECTION 2. TECHNICAL APPROACH

This section discusses in greater detail the sampling and analytical procedures Entropy will use. Table 2-1 at the end of this section summarizes the number of samples that will be taken and the procedures that will be used for sample collection and analysis.

### 2.1 NON-FLUE GAS SAMPLES.

In the interest of safety, Entropy prefers that non-flue gas samples (waste feeds and scrubber makeup and discharge) be collected by personnel familiar with the incinerator and its operation. If Eli Lilly cannot provide manpower to collect these samples, this manpower can be supplied by Entropy.

Sample collection methods for waste feed and scrubber makeup and discharge samples should generally follow the techniques described in Sampling and Analysis Methods for Hazardous Waste Incineration (First Edition), a guideline document prepared for the EPA by Arthur D. Little, Inc. Specific collection methods to be employed are discussed below. Entropy will provide equipment and guidance in the collection of these samples.

#### 2.1.1 Waste Materials.

Primary and secondary waste feed samples will be collected three times during each run. The three samples collected will be of equal volume. Samples of each waste will be combined to provide one primary and one secondary waste feed sample representing the entire run.

Each run sample will be homogenized, and aliquots taken for analysis for POHC's and organically bound chlorine. The remainder of each sample will be held in the event further analysis is needed. Analysis for the parameters of interest will be performed according to the methods shown in Table 2-1.

**ENTROPY**

### 2.1.2 Scrubber Makeup and Discharge Samples.

Scrubber makeup and discharge samples will be collected three times during each test run. The three samples collected will be of equal volume. Samples of each type will be mixed to provide one makeup sample and one discharge sample representing each run.

Scrubber makeup samples will be analyzed for the designated POHC's. Scrubber discharge samples will be analyzed for POHC's and pH. Table 2-1 gives the analytical procedures to be followed in making these determinations.

## 2.2 FLUE GAS SAMPLES.

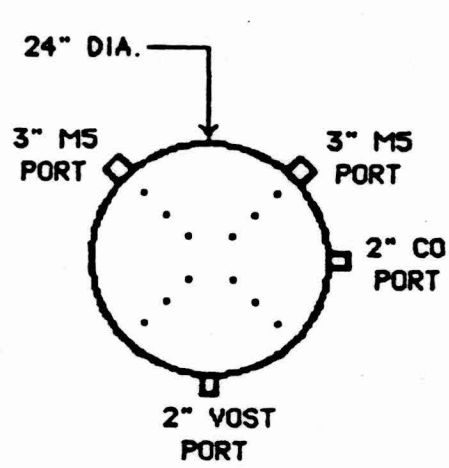
Entropy will supply all equipment, materials, and labor for the collection and analysis of all flue gas samples. The following sections describe the individual sampling methods in detail.

### 2.2.1 Exhaust Gas Flow Rate and Excess Air.

EPA Methods 1 and 2 will be used to determine sampling point locations and flue gas velocity and flow rate, respectively. Figure 2-1 is a dimensional diagram showing the sampling location and stack traverse points. The type S pitot tubes will conform to the geometric specifications outlined in EPA Method 2, and will be assigned a coefficient of 0.84. Differential pressures will be measured with magnehelic gauges of appropriate range or with fluid manometers. Chromel-alumel thermocouples will be used for the measurement of stack gas temperatures.

EPA Method 3 will be used for flue gas composition and excess air determinations. Constant rate sampling will be used to obtain an integrated sample of stack gas. A peristaltic pump will be used to deliver 500 ml/min of stack gas to a Tedlar bag. Analysis will be by orsat apparatus, following the procedures of EPA Method 3. Determinations of the moisture content of the flue gases will follow the procedures of EPA Method 4.

**ENTROPY**

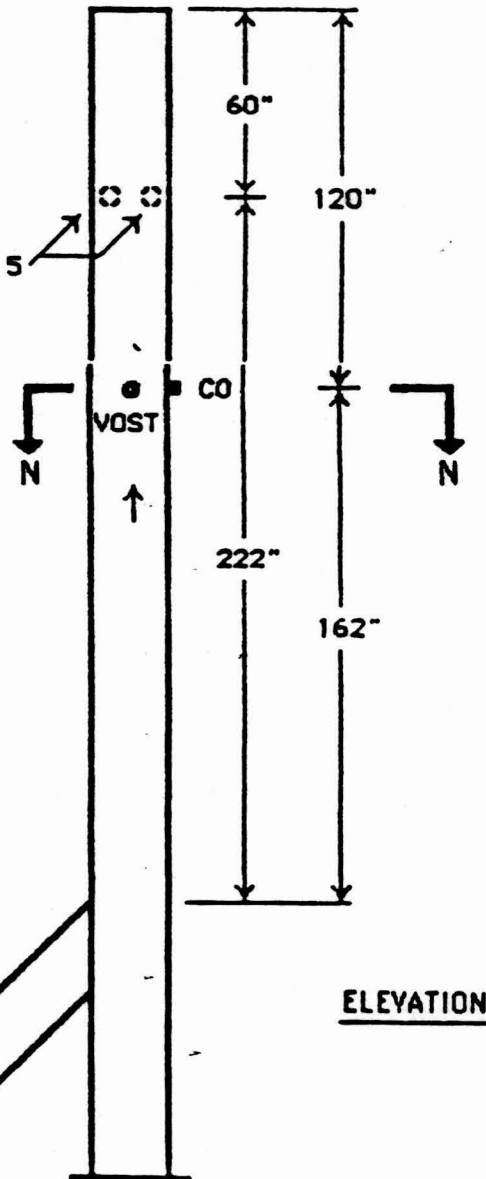


**METHOD 5  
TRAVERSE POINTS**

2 AXES  
6 POINTS/AXIS  
12 TOTAL POINTS

**SECTION N-N**

PROPOSED METHOD 5  
TEST PORTS



**ELEVATION**

FIGURE 2-1. INCINERATOR STACK TEST LOCATION SHOWING TRAVERSE POINTS

A cyclonic flow pattern is not expected in this stack. However, the EPA requests that Entropy measure the yaw angle using the procedure described in EPA Method 2.

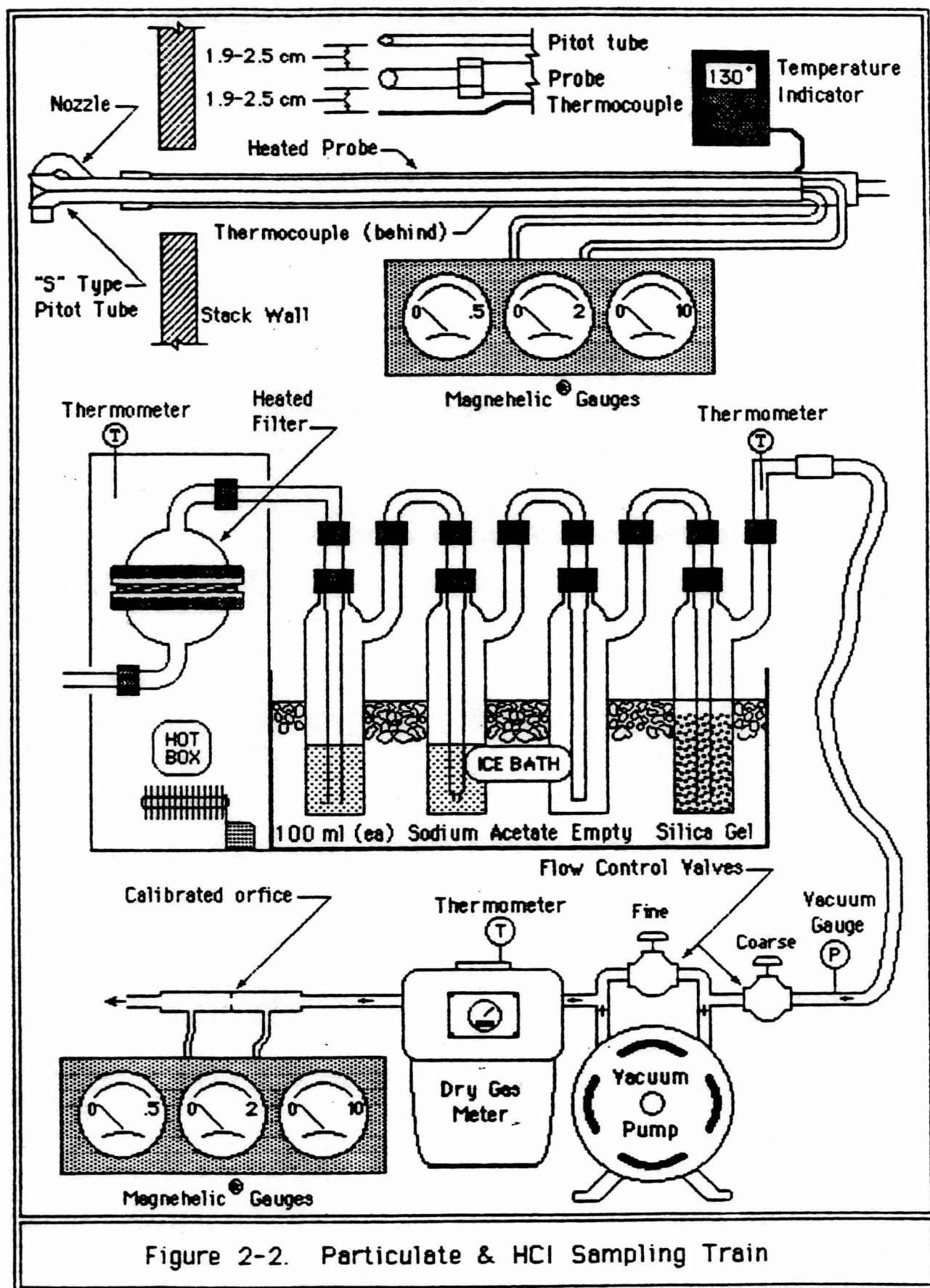
#### 2.2.2 Exhaust Gas Particulate and HCl.

EPA Reference Method 5 will be used to determine the concentration of particulate matter in the incinerator effluent gas. The Method 5 train will be modified to capture chlorides as well. The modification will consist of replacing the water in the first two impingers with 0.1 N sodium acetate. The third impinger will remain empty, and the fourth will contain silica gel. The impinger portion of this train is depicted in Figure 2-2.

Each particulate test run will be at least 120 minutes in duration, and a minimum of 30 dry standard cubic feet will be collected during each run. The average sampling rate for each run will be within  $\pm 10\%$  of 100% isokinetic conditions. Reference Method 5 analytical procedures will be strictly adhered to in the determination of particulate emissions. The impinger catch will be analyzed for chlorides by ion chromatography.

#### 2.2.3 Exhaust Gas POHC'S.

Concentrations of POHC's in the stack effluent will be measured using the Volatile Organic Sampling Train (VOST) concept. The VOST system is designed to draw effluent gas through a series of sorbent traps. The first trap will contain Tenax resin, and the second and third traps will contain a section of Tenax followed by a section of activated charcoal. A third trap has been added to the VOST system in order to evaluate breakthrough. The Tenax trap will be preceded by a water-cooled condenser arranged so that condensate will drain through the Tenax. The VOST sampling train is depicted schematically in Figure 2-3.



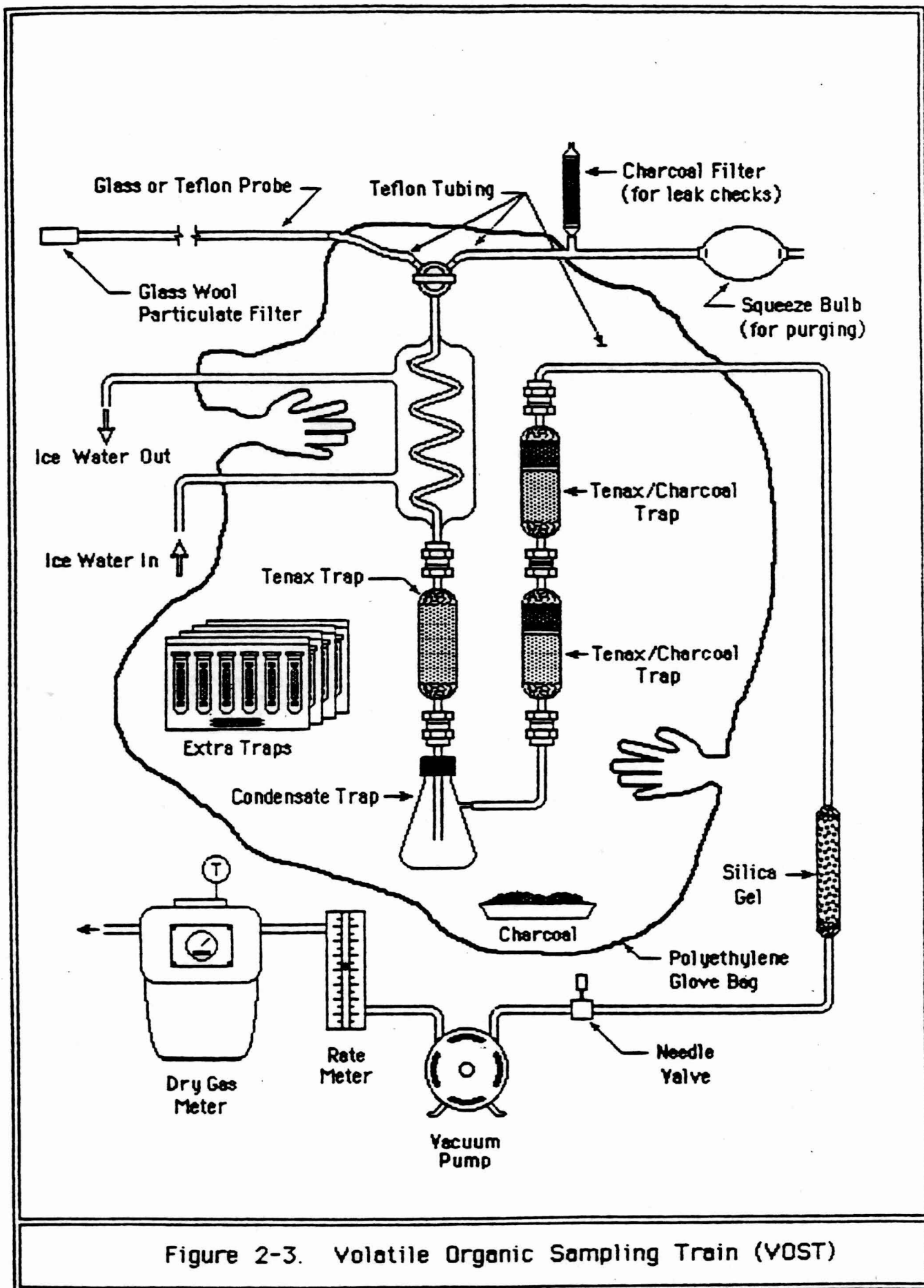


Figure 2-3. Volatile Organic Sampling Train (VOST)

Sampling will take place for 120 minutes per run and will be performed simultaneously with the EPA Method 5 train. Three runs will be performed for the trial burn. Each pair of traps will be replaced every 40 minutes so that three pairs of traps will be collected per run, nine pairs per condition. The VOST will be operated at a sampling rate of 0.5 liter per minute (20 liters per sample).

Analysis for POHC's will follow the guidelines of EPA Method 624, a method that has been proposed for organic compounds that are amenable to the purge-trap-desorb (P-T-D) technique. The organic contents of one pair of Tenax and charcoal traps will be thermally desorbed into water using a carrier gas. The desorbed compounds will then be purged from the water and collected on an analytical trap containing Tenax and charcoal. The compounds are then back-purged off the trap into the GC/MS. Nitrogen will be the carrier gas in all cases.

Table 2-1

TRIAL BURN SAMPLING AND ANALYSIS PROCEDURES

<u>Matrix</u>	<u>Parameter</u>	<u>Sampling Method</u>	<u># of Samples</u>	<u># of Analyses</u>	<u>Analysis Method</u>
Exhaust Gas	POHC's	VOST	9	9	EPA Method 624 (GC/MS)
Exhaust Gas	CO	EPA Method 10	----continuous----		EPA Method 10 (Byron 233A)
Exhaust Gas	CO <sub>2</sub> , O <sub>2</sub>	EPA Method 3	3	3	EPA Method 3 (Orsat)
Exhaust Gas	Particulate	EPA Method 5	3	3	EPA Method 5 (gravimetric)
Exhaust Gas	HCl	EPA Method 5	3	3	Ion Chromatography
Waste Mixture	CCl <sub>4</sub>	Grab Samples	9	3	GC/ECD
Waste Mixture	CH <sub>2</sub> Cl <sub>2</sub>	Grab Samples	9	3	GC/FID
Waste Mixture	Chlorine	Grab Samples	9	3	ASTM D2361-66
Scrubber Makeup	CCl <sub>4</sub>	Grab Samples	9	3	EPA Method 501.2
Scrubber Makeup	CH <sub>2</sub> Cl <sub>2</sub>	Grab Samples	9	3	8010 (SW-846)
Scrubber Discharge	CCl <sub>4</sub>	Grab Samples	9	3	EPA Method 501.2
Scrubber Discharge	CH <sub>2</sub> Cl <sub>2</sub>	Grab Samples	9	3	8010 (SW-846)
Scrubber Discharge	pH	Grab Samples	9	3	EPA Method 150.1



### SECTION 3. QUALITY ASSURANCE

#### 3.1 EQUIPMENT INSPECTION AND MAINTENANCE.

Each item of field test equipment purchased by Entropy is assigned a unique, permanent identification number. An effective preventive maintenance program is necessary to ensure data quality. Each item of equipment returning from the field is inspected before it is returned to storage. During the course of these inspections, items are cleaned, repaired, reconditioned, and recalibrated where necessary.

Each item of equipment transported to the field for this test program is inspected again before being packed to detect equipment problems which may originate during periods of storage. This minimizes lost time on the job site due to equipment failure.

Occasional equipment failure in the field is unavoidable despite the most rigorous inspection and maintenance procedures. For this reason, Entropy routinely transports to the job site replacement equipment for all critical sampling train components.

#### 3.2 EQUIPMENT CALIBRATION.

New items for which calibration is required are calibrated before initial field use. Equipment whose calibration status may change with use or with time is inspected in the field before testing begins and again upon return from each field use. When an item of equipment is found to be out of calibration, it is repaired and recalibrated or retired from service. All equipment is periodically recalibrated in full, regardless of the outcome of these regular inspections.

Calibrations are conducted in a manner, and at a frequency, which meets or exceeds U. S. EPA specifications. Entropy follows the calibration procedures outlined in the EPA Reference Methods, and those recommended within the Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III (EPA-600/4-77-027b, August, 1977). When

the Reference Methods are inapplicable, Entropy uses methods such as those prescribed by the American Society for Testing and Materials (ASTM).

Data obtained during calibrations are recorded on standardized forms, which are checked for completeness and accuracy by the quality assurance manager or the quality assurance director. Data reduction and subsequent calculations are performed using Entropy's own computer facilities. Calculations are checked at least twice for accuracy. Copies of calibration forms are included in the test or project reports.

Emissions sampling equipment requiring calibration includes pitot tubes, pressure gauges, thermometers, flow meters, dry gas meters, and barometers. The following sections elaborate on the calibration procedures followed by Entropy for these items of equipment. Calibration data for the sampling equipment will be provided to the EPA on site just prior to the trial burn testing.

### 3.2.1 Pitot Tubes.

All Type S pitot tubes used by Entropy, whether separate or attached to a sampling probe, are constructed in-house or by Nutech Corporation. Each new pitot is calibrated in accordance with the geometry standards contained in EPA Reference Method 2. A Type S pitot tube, constructed and positioned according to these standards, will have a coefficient of  $0.84 \pm 0.02$ . This coefficient should not change as long as the pitot tube is not damaged.

Each pitot tube is inspected visually upon return from the field. If a cursory inspection indicates damage or raises doubt that the pitot remains in accordance with the EPA geometry standards, the pitot tubes are refurbished as needed and recalibrated.

### 3.2.2 Differential Pressure Gauges.

Some meter consoles used by Entropy are equipped with 10-in. water column (W.C.) inclined-vertical manometers. Fluid manometers do not require calibration other than leak checks. Manometers are leak-checked

**ENTROPY**

in the field prior to each test series, and again upon return from the field.

Most of Entropy's meter consoles are equipped with Magnehelic differential pressure gauges. Each set of gauges is calibrated initially over its full range, 0-10 inches W.C. After each field use, the calibration of the gauge set is checked against an inclined manometer at the average delta p encountered during the test. If the agreement is within  $\pm 5$  percent, the calibration is acceptable.

### 3.2.3 Thermometers.

3.2.3.1 Impinger Thermometer. On site, prior to the start of testing, the thermometer used to monitor the temperature of the gas leaving the last impinger is compared with a mercury-in-glass thermometer which meets ASTM E-1 No. 63F specifications. The impinger thermometer is adjusted if necessary until it agrees within  $2^{\circ}\text{F}$  of the reference thermometer. If the thermometer is not adjustable, it is labeled with a correction factor.

3.2.3.2 Dry Gas Meter Thermometer. The thermometer used to measure the temperature of the metered gas sample is checked prior to each field trip against an ASTM mercury-in-glass thermometer. The dry gas meter thermometer is acceptable if the values agree within  $\pm 5.4^{\circ}\text{F}$ . Thermometers not meeting this requirement are adjusted or labeled with a correction factor.

3.2.3.3 Stack Temperature Sensor. All thermocouples employed by Entropy for the measurement of flue gas temperatures are calibrated upon receipt. Initial calibrations are performed at three points (ice bath, boiling water, and hot oil). An ASTM mercury-in-glass thermometer is used as a reference. The thermocouple is acceptable if the agreement is within 1.5 percent (absolute) at each of the three calibration points.

On site, prior to the start of testing, the reading from the stack gas thermocouple-potentiometer combination is compared with an ASTM

mercury-in-glass reference thermometer. If the two agree within  $\pm 1.5$  percent (absolute), the thermocouple and potentiometer are considered to be in proper working order for the test series.

After each field use, the thermocouple-potentiometer system is compared with an ASTM mercury-in-glass reference thermometer at a temperature within  $\pm 10$  percent of the average absolute stack gas temperature. If the absolute temperatures agree within  $\pm 1.5$  percent, the temperature data are considered valid.

### 3.2.4 Dry Gas Meter and Orifice Meter.

Two procedures are used to calibrate the dry gas meter and orifice meter simultaneously. The full calibration is a complete laboratory procedure used to obtain the calibration factor of the dry gas meter. Full calibrations are performed over a wide range of orifice meter settings. A simpler procedure, the posttest calibration, is designed to check whether the calibration factor has changed. Posttest calibrations are performed after each field test series at an intermediate orifice meter setting (based on the test data) and at the maximum vacuum reached during the test.

Entropy uses as a transfer standard a dry gas meter that is calibrated annually against a spirometer. During the annual calibration, triplicate calibration runs are performed at seven flow rates ranging from 0.25 to 1.40 cfm.

3.2.4.1 Dry Gas Meter. Each metering system receives a full calibration at the time of purchase and a posttest calibration after each field use. If the calibration factor, Y, deviates by less than five percent from the initial value, the test data are acceptable. If Y deviates by more than five percent, the meter is recalibrated and the meter coefficient (initial or recalibrated) that yields the lowest sample volume for the test runs is used.

EPA Reference Method 5 requires another full calibration anytime the posttest calibration check indicates that Y has changed by more than five

**ENTROPY**

percent. Standard practice at Entropy is to recalibrate the dry gas meter anytime  $Y$  is found to be outside the range  $0.98 \leq Y \leq 1.02$ .

3.2.4.2 Orifice Meter. An orifice meter calibration factor is calculated for each flow setting during a full calibration. If the range of values does not vary by more than 0.15 in.  $H_2O$  over the range of 0.4 to 4.0 in.  $H_2O$ , the arithmetic average of the values obtained during the calibration is used.

### 3.2.5 Barometer.

Each field barometer is adjusted before each test series to agree within  $\pm 0.1$  inches of a reference aneroid barometer. The reference barometer is checked weekly against the station pressure value (corrected for elevation difference) reported by the National Weather Service station at the Raleigh-Durham airport, approximately 2.5 miles from Entropy's location.

### 3.3 SAMPLE CUSTODY.

Samples are collected, transported, and stored in clean containers which are constructed of materials inert to the analytical matrix. Only containers which allow air tight seals are used. When necessary, containers are employed which inhibit photochemical reactions.

All sample containers are labelled with the following information:

- (a) condition number
- (b) test set number
- (c) a code indicating the test method

Additional information relating to the sample is recorded on the data sheet for the associated sampling run. Accordingly, the sampling data sheet contains all the information listed above, plus the date and time the sample is acquired and supplemental information such as observations

**ENTROPY**

pertinent to the quality of the sample. For samples in liquid media, the sample levels are marked on the outside of the container to indicate loss of sample.

Samples are stored for transport from the field to the laboratory in storage boxes constructed in a fashion which minimizes movement and prevents breakage of containers. For example, boxes used for transporting glass containers have foam inserts with form-fitting cutouts. Sample transport boxes are always locked except when in use. Vans containing equipment and samples are locked whenever they are left unattended.

Samples remained in the custody of the sampler from the time of sample acquisition until custody is transferred to the laboratory analyst. All custody transfers are documented on the chain-of-custody form initiated by the sampler at the time of sample collection in the field. This form remained with the sample at all times.

Analytical data are recorded and identified in a manner similar to that for the sampling data. All data generated from the analysis of samples are documented with the following information:

- (a) source identifier
- (b) sample run identifier
- (c) analyte identifier
- (d) sample matrix identifier
- (e) analyst identifier
- (f) analysis date

Portions of samples remaining after analysis are returned to their original sample containers. These samples are stored in designated storage areas until their destruction is authorized.

### 3.4 SAMPLING AND ANALYTICAL SENSITIVITY.

The sensitivities of the proposed sampling and analysis techniques must be sufficient to determine Destruction and Removal Efficiencies with the required precision. Typically, the lower detectable limit for organic compounds is below 100 nanograms (ng) per sample, and often as low as 10 ng. The candidate POHC in this trial burn to which the analytical apparatus has the lowest sensitivity is carbon tetrachloride.

The calculations in Table 3-1 show that, with a DRE of 99.99%, the proposed feed rates and POHC concentrations will yield samples of sufficient size for the required analytical accuracy.

TABLE 3-1  
ESTIMATED MASS OF POHC PER SAMPLE

<u>Compound</u>	<u>#/hr fed</u> <sup>1</sup>	<u>g/hr out</u> <sup>2</sup>	<u>ug/liter</u> <sup>3</sup>	<u>ug/sample</u> <sup>4</sup>
Methylene Chloride	335.25	15.24	1.49	29.9
Carbon Tetrachloride	80.4	3.66	0.38	7.53

<sup>1</sup> based on primary waste feed rate

<sup>2</sup> based on 99.99% DRE

<sup>3</sup> based on 6,000 scfm

<sup>4</sup> based on 20 liter sample per pair of VOST tubes



### 3.5 METHOD 5.

#### 3.5.1 Sampling.

During setup of the Method 5 train, tweezers are used to place a preweighed filter in the filter holder. The filters are properly centered to prevent the sample gas stream from bypassing the filter and to ensure a leak-free system. 100 mL of 0.1 N sodium acetate is placed in each of the first two impingers to capture the HCl emissions. The third impinger is left empty, and a preweighed amount of silica gel (with container) of approximately 200 g is placed in the fourth impinger. Temperature sensors are placed on the Method 5 train to measure the stack, filter, and impinger exit temperatures.

At the sampling location, the filter and probe heating systems are activated and set at the designed operating temperatures. Time is allowed for temperatures to stabilize. To maintain isokinetic sampling rates, a nozzle size used for sampling is determined from preliminary velocity measurements and moisture predictions. The minimum number of sampling points is selected according to EPA Method 1.

A pretest leak-check on the system is performed by plugging the nozzle and pulling a vacuum of 15 inches of Hg with an allowable leakage rate of 0.02 dscfm. To return the system to atmospheric pressure after leak-checking, the plug is slowly removed from the nozzle. The slow removal of the plug prevents the back-flushing of reagents within the impingers. A leak-check is also performed on the pitot tubes. After the filter and probe heating systems are maintained at the desired operating temperature, all ports are cleaned out, and the nozzle covering is removed. Upon insertion of the probe to the first traverse point, ports are plugged to prevent the introduction of ambient air into the gas stream. Crushed ice is placed around the impingers to keep the impinger exit temperature below 20°C.

Sampling is kept at  $\pm 10\%$  isokinetics by adjusting the sampling rate of the meterbox pump based on the gas flow rate at the sampling point in the stack. This is accomplished by using a nomgraph designed for the use with



the Type S pitot tube having a coefficient of  $0.84 \pm 0.02$  and stack gas with a dry molecular weight of  $29 \pm 4$ . In addition to Method 5, EPA Method 2 is used to determine the stack pressure, temperature, and range of velocity heads. EPA Method 3 sampling is used for molecular weight determination, and EPA Method 4 is used to determine the stack gas moisture content. Adjustments are continuously made to the filter heating system to maintain a temperature of  $120 \pm 14^{\circ}\text{C}$ .

At the conclusion of each run a posttest leak-check is also performed. The leak-check is conducted at a vacuum equal to, or greater than, the maximum value recorded during the sample run with an acceptable leakage rate of 0.02 dscfm.

### 3.5.2 Sample Recovery.

In an area sheltered from wind and dust, the sealed particulate sample filter holder is opened and the filter and all loose particulate are transferred to a 500 mL glass jar with a Teflon-lined screw cap. All filter handling is performed with tweezers. Any filter material adhering to the filter gasket is removed with a sharp blade or a clean nylon bristle brush and placed in the sample container. The container is then sealed, labeled with the run number, and placed in a locking sample transport box designed to hold samples from three runs. The outside of the transport box is labeled with the plant name, sampling location, run numbers, date of collection, sample type, and initials of the sample collector.

The sample-exposed half of the filter holder is brushed and rinsed with acetone into a 500 mL glass jar with Teflon-lined cap. Removal of all adhering particulate is confirmed by visual inspection. The outside of the sealed probe and nozzle are wiped clean of loose particulate. The nozzle is removed and brushed with acetone and a nylon bristle brush until no particles are visible in the rinse. The rinsings are added to the filter holder acetone rinse.

Probe cleaning is always performed by two people. The probe is cleaned by tilting it and squirting acetone into the upper end while brushing with a

nylon bristle brush using a rotating motion. The acetone rinse container is positioned at the lower end to catch the drainage. Brushing and rinsing is continued until no visible particulate remained in the rinse. The brush is rinsed into the acetone sample container, which is sealed, marked with the liquid level, labeled with the run number, and placed in the locking transport box.

Standard procedure at Entropy is to measure the liquid in the impingers at Entropy's laboratory, rather than in the field. At the conclusion of each sampling run, the contents of the first three impingers are transferred quantitatively to the same 1000 mL glass jar in which the original 200 mL of sodium acetate is transported to the field. The liquid level is marked on the outside of the container, and the container is sealed with a Teflon-lined screw cap, labeled with the run number, and placed in the locking sample transport box.

The impingers are rinsed three times with distilled, deionized water. The impinger rinse is collected in a separate container, which is marked for liquid level, labeled with the run number, and placed in a locking sample transport box.

Entropy uses indicating silica gel which changes from blue to pink as it gains moisture. At the end of the run, a visual estimate of the percentage of the silica gel which appeared spent is noted on the sample chain of custody form. The silica gel is transferred to its original container and sealed. The container is labeled with the run number and returned to the locking sample transport box.

### 3.5.3 Analyses.

3.5.3.1 Particulate. The analytical procedures associated with this method consist of evaporations and weighings. Although these procedures are relatively simple, it is essential that sample handling be minimized and be done carefully to avoid sample contamination or loss.

The dry filter and loose particulate matter in the sample container are transferred to a tared beaker. Particles adhering to the sample container

are rinsed with acetone, with brushing if necessary, into the sample container containing the acetone rinses from the nozzle, probe, and filter holder front half. The beaker containing the filter and dry particulate is desiccated for a minimum of 24 hours, then weighed on a Sartorius Model 2442 analytical balance until a constant weight is achieved.

The term "constant weight" means a difference between two consecutive weighings of  $\leq 0.5$  mg or 1% of the particulate weight, whichever is greater. A minimum of 6 hours of desiccation must separate consecutive weighings. Class-S standard weights are used to check the span of the balance before each series of weighings. The same balance is used for all weighings. Sample weights are reported to the nearest 0.1 mg. Clean disposable gloves are worn during any handling of tared sample beakers.

The contents of these sample containers are measured volumetrically and transferred to separate tared beakers. Evaporations are performed over a Bockel steam bath, with the solvent temperatures kept below their boiling points to prevent "bumping." At Entropy, the steam bath is situated inside a Hemco high volume fume hood to prevent vapor concentration and expedite evaporation. The fume hood is equipped with a filtered air inlet to keep foreign matter out of the samples.

After each beaker had been evaporated to dryness, it is moved to a desiccator for a minimum of 24 hours and weighed to a constant weight. The weight is reported to the nearest 0.1 mg. All beaker handling is done with clean disposable gloves.

Each blank is evaporated to dryness, desiccated, and weighed to a constant weight in exactly the same fashion as the sampling train rinses. In no case is a blank value of more than 0.01 mg/g subtracted from the sample weight.

The volume of liquid in the impinger water jar is measured to the nearest 1 mL with a graduated cylinder. The volume is recorded on the laboratory reporting form. The water is then returned to its original container to await further analysis.

The weight of the silica gel (with container) is determined to the nearest 0.5 mg using a triple-beam balance and reported on the laboratory form. The spent silica gel is then discarded.

**ENTROPY**

3.5.3.2 Hydrogen Chloride (HCl). The analyses for HCl is performed by use of ion chromatography. The ion chromatograph is calibrated at the beginning of each shift or sample batch using not less than two standards of concentration which overlap the analytical range. In addition, the instrument is recalibrated whenever any change is made in the operating parameters (i.e., columns, eluent, regenerant).

Eluent blanks are analyzed after every fifth injection and whenever eluent is changed to insure that no cross contamination or carryover has occurred.

Method blanks are prepared and analyzed in the same manner as samples except that the glassware used in extraction did not contain any sample matrix (e.g., no soil or water). The blank is designed to monitor the background generated only from the glassware. Method controls are similar to blanks except that a non-contaminated matrix is added to the glassware. Lab method controls used a matrix generated from the lab (e.g., distilled water), whereas field controls used a matrix from a non-contaminated area (e.g., water or soil taken from off-site). Control samples are used to monitor the background generated from the matrix being analyzed. Method blanks and controls are analyzed with every ten samples or every batch, whichever is more frequent.

Every 10th sample is split and analyzed in duplicate from start to finish as a separate sample. Duplicate injections are made on every fifth injection to verify reproducibility. This procedure is not waived for autoinjectors.

At every 10th sample, a spiked recovery is performed to determine the recovery efficiency. This spike is either a control sample or an actual sample with a known amount of standard added. If an actual sample is used, it is split; one-half is spiked and the other half is analyzed for background.

### 3.6 VOST.

Entropy will follow the procedures outlined in "Protocol for the Collection and Analysis of Volatile POHC's Using VOST" in the collection and analysis of all samples. This section addresses some quality assurance considerations pertinent to the proposed trial burn sampling and analysis.

#### 3.6.1 Sampling.

All sealed tenax cartridges stored in glass culture tubes are placed into Ziploc freezer bags with an open NIOSH type charcoal tube inside each bag. (Figure 5.2). The purpose of this charcoal tube is to scavenge any hydrocarbons that might penetrate through the freezer bag. The bagged tubes are prepared for shipment by placing them into coolers containing sealed cold packs and another open charcoal tube. The cooler is kept dry to prevent the possible contamination of the tubes by the contents of the water. One pair of the blank cartridges is designated as a trip blank and included with the shipment of cartridges to the test site. If contamination had been suspected, this pair of cartridges would have been analyzed to monitor potential contamination during storage and shipment.

Prior to setup, all glassware and sample transfer lines are rinsed with 0.1 N nitric acid followed by a rinsing with distilled water. The sample transfer lines are Teflon with Teflon swagelock fittings capable of forming leak-free, vacuum-tight connections without the use of sealing grease. The sample collection parts of the VOST are placed inside a polyethylene Atmos glove bag along with all the necessary tubes (still in Ziploc bags) needed for that sampling day. A petri dish of charcoal is also placed in the Atmos glove bag before sealing. The probe is a 3-foot length Teflon tubing with glass wool placed in the open end to prevent particulate from entering the sample train. A cartridge containing silica gel is placed between the VOST and the metering system to remove any remaining moisture before the sample gas entered the dry gas meter.

Following setup, a pretest leak check is performed on the VOST by pulling a vacuum of approximately 25 to 30 inches of Hg with an allowable leakage rate of 0.1 inches of Hg per minute. After leak checking, the train is returned to atmospheric pressure by opening the isolation valve to introduce ambient air into the train through a charcoal tube. This procedure should have minimized contamination of the VOST from fugitive emissions at the sampling location.

The sampling port for the VOST is plugged to prevent the introduction of ambient air after placing the probe into the stack. Prior to sampling, the probe is purged with stack gas by use of a squeeze bulb. During testing, tubes are removed from and replaced into the freezer bag as needed. Also one pair of field blanks are prepared each day during sampling to assess any contamination at the test site. Cold water is circulated through the condensers to cool the sample gas stream below 20°C. Following each test run, a posttest leak check is performed by pulling a vacuum of approximately 10 inches greater than the operating pressure.

### 3.6.2 Sample Recovery.

Cleanup of the VOST involved rinsing of the probe with distilled water which flowed through the first sorbent cartridge and into the condensate trap. Therefore any carbon tetrachloride and methylene chloride which may have condensed within the probe should have been captured by the tenax. The captured condensate is poured into a 50 mL graduated cylinder. The trap is then rinsed with distilled water three times and combined with the condensate. Distilled water is added to the graduated cylinder until the total volume reached 50 mL. After thorough mixing of the condensate a 40 mL aliquot is taken. At the end of the testing day the Atmos glove bag is opened, and the cartridges (sealed in their culture tubes and Ziploc bags) are removed and placed back into the cooler with fresh cold packs and open charcoal tubes.



### 3.6.3 Analyses.

A purge and trap desorption device (P-T-D) meeting all the criteria of section 10.2 of "Protocol for Analysis of Sorbent Cartridges from the Volatile Organics Sampling Train" and section 5.2 of USEPA Method 624 is used for the analysis of carbon tetrachloride. This unit (Nutech Model 8522) is modified by the installation of a secondary thermal desorption chamber (Clamshell Oven) which accommodates the Tenax and Tenax/Charcoal sorbent cartridges. The P-T-D is interfaced with a GC/MS (Hewlett Packard 5895) which is tuned to meet all the criteria of EPA Method 624 for a 50 ng injection of 4-Bromofluorobenzene (BFB).

Standards and internal standards are loaded onto pairs of cartridges using flash evaporation which employs nitrogen carrier at 40 mL/min. in the same flow direction as sample collection. Stock standards are prepared by weighing pure reference materials into methanol and diluting such that injections of 1 to 3 ul would give loadings which bracket the working range of the P-T-D GC/MS system.

3.6.3.1 Breakthrough. To evaluate breakthrough, a third sorbent trap has been added to the VOST system. The first and second traps shall be analyzed together, and third sorbent trap shall be analyzed separately. According to the EPA, breakthrough has occurred if the amount of POHCs measured on the third sorbent trap exceeds 10% of the total sample catch. Following the analysis of the first VOST sample, if breakthrough is not apparent, only the first and second sorbent traps will be analyzed for the remaining samples. However, if breakthrough is apparent, the EPA may question the validity of the results.

3.6.3.2 Condensate. The VOST condensate collected during the first run shall be analyzed for the designated POHCs. If the concentrations within the condensate exceeds 25% of the total sample, it will be required that all condensate samples be analyzed. However, none of the remaining condensate samples will be analyzed if concentrations do not exceed 25% of the total sample.

**ENTROPY**

### 3.7 SCRUBBER WATER ANALYSES.

All methylene chloride values will be obtained using SW-846 sample introduction technique 5030 (purge and trap) with the analysis by method 8010 (Halogenated Volatiles). The analytical column will be 6' X 1/8" SS packed with 1% SP1000 on 60/80 mesh carbopak B. An FID detector will be used.

Carbon tetrachloride values will be obtained using EPA method 501.2 (liquid liquid extraction with iso-octane). The analytical column will be 10% SP1000 on 100/120 mesh Supelcoport packed in 10' X 1/8" nickel. Detection is achieved using the electron capture detector.

### 3.8 LIQUID SAMPLE STORAGE AND TRANSPORT.

Due to the volatility of some POHC's, care will be taken to prevent loss of POHC's from collected samples of scrubber makeup and scrubber effluent. Sample containers will be filled completely, with no head space above the liquid. All sample containers will be stored and transported in insulated chests with cold packs (no water). Samples will be maintained at 4°C or less until the time of analysis.

### 3.9 SORBENT CARTRIDGE BLANKS.

Additional sorbent cartridges for the VOST will be taken to the sampling site to serve as field and trip blanks. Cartridges designated as field blanks will be uncapped and exposed to the atmosphere surrounding the respective train for the length of time required to exchange the cartridges in the train. Field blanks will be capped and stored for transport in the same manner as sample-exposed cartridges. Additional cartridges, designated as trip blanks, will be transported to and from the field and otherwise treated as the other cartridges, except that the caps will not be removed.



JAN 19 1987

# ENTROPY

ENVIRONMENTALISTS INC.

POST OFFICE BOX 12291  
RESEARCH TRIANGLE PARK  
NORTH CAROLINA 27709-2291  
919-781-3550

January 14, 1987

Mr. Ron Pitzer  
Eli Lilly and Company  
Lilly Corporate Center  
Indianapolis, Indiana 46285

Dear Mr. Pitzer:

In response to the NOD's, I've enclosed a letter from Oxford Laboratories, Inc. addressing question 4. SW-846 Method 8010 will be used for the analysis of the POHCs, carbon tetrachloride and methylene chloride, in the waste feed. Scrubber samples will be analyzed for the POHCs according to SW-846 Method 5030.

Scrubber makeup and discharge samples will be collected in "VOA" vials and composited in the laboratory just prior to analysis. This compositing procedure should satisfy the agency's concern over the loss of the volatile POHCs.

Oxford Laboratories is presently gathering the information concerning the QA/QC for the waste feed and scrubber water analyses. I will forward this information to you as soon as possible.

If there is anything else I can provide, please call me.

Sincerely,

*Tony Wong*  
Tony Wong  
Assistant Sales Director

TW/rlo

Enclosure

Oxford Laboratories, Inc.

Analytical and Consulting Chemists  
1316 South Fifth Street  
Wilmington, N.C. 28401  
(919) 763-9793

January 8, 1987

Entropy  
P.O. Box 12291  
Research Triangle Park, N.C.  
27709-2291

Attention: Tony Wong

Dear Mr. Wong,

Please note that the correct method code for the analysis of Methylene chloride and Carbon tetrachloride for samples within your Eli Lilly project is SW846 Method 8010 (Volatile halogenated organics). This method employs gas chromatography with Hall Electrolytic conductivity detector.

Primary waste feed samples will be analyzed by direct injection after dilution with methanol. Aqueous samples such as scrubber discharges and scrubber make-up waters will be analyzed using the purge and trap technique (method 5030).

If composite sample from waste streams is required, primary waste feeds can be composited by blending individual grab samples in the field. But in the case of aqueous samples containing trace levels of the volatiles, individual grab samples should be collected in duplicate using 40ml "VOA" vials.

All of the individual grab samples which represent a given stream would then be composited in the laboratory just prior to analysis. This procedure will minimize losses of the analytes due to volatilization and will prevent cross contamination from high-level samples such as primary feeds.

The VOA samples should be stored and shipped separately from high level samples. It is recommended that several field blanks be transported with these samples to monitor for cross contamination.

Sincerely,

  
Bruce A. Babson

BAB/lo

A P P E N D I X    I I

*Lilly*

**Eli Lilly and Company**

307 East McCarty Street  
Indianapolis, Indiana 46285  
(317) 261-2000

March 31, 1986

CERTIFIED MAIL  
RETURN RECEIPT REQUESTED

Environmental Quality Board  
P.O. Box 11488  
Santurce, Puerto Rico 00910-1488

Attention: Chairman, Environmental  
Quality Board

Dear Sir:

I am the chief financial officer of Eli Lilly and Company, Lilly Corporate Center, Indianapolis, Indiana 46285. This letter is in support of the use of the financial test to demonstrate financial responsibility for liability coverage and closure and/or post-closure care, as specified in Rules I-806 and II-808.

The owner or operator identified above is the owner or operator of the following facilities for which liability coverage is being demonstrated through the financial test and/or corporate guarantee specified in Rules I-806 and II-808.

<u>Identification No.</u>	<u>Name/Address</u>	
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	Financial Test
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	Financial Test
IND 006421325	Eli Lilly and Company Materials Center 1402 South Dakota Street Indianapolis, Indiana 46285	Financial Test

PRD 091024786

Eli Lilly Industries, Inc      Corporate.  
Mayaguez Plant                      Guarantee  
Road #2, KM 146.7  
Mayaguez, Puerto Rico 00708

1. The owner or operator identified above owns or operates the following facilities for which financial assurance for closure or post-closure care is demonstrated through the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by the test are shown for each facility:

<u>Iden. No.</u>	<u>Name / Address</u>	<u>Closure Cost (\$)</u>
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	\$562,000
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	569,000
IND 006421325	Eli Lilly and Company Materials Center 1402 S. Dakota Street Indianapolis, Indiana 46285	73,800

2. The owner or operator identified above guarantees, through the corporate guarantee specified in Rules I-806 and II-808, the closure and post-closure care of the following facilities owned or operated by its subsidiaries. The current cost estimates for the closure or post-closure care so guaranteed are shown for each facility:

PRD 091024786	Eli Lilly Industries, Inc. Mayaguez Plant Road #2, KM 146.7 Mayaguez, Puerto Rico 00708	\$165,500
---------------	--	-----------

3. Where the Commonwealth of Puerto Rico is not administering the financial requirements of Rules I-806 and II-808, this owner or operator is demonstrating financial assurance for the closure or post-closure care of the following facilities through the use of a test equivalent or substantially equivalent to the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by such a test are shown for each facility:  
NONE

4. The owner or operator identified above owns or operates the following hazardous waste management facilities for which financial assurance for closure or, if a disposal facility, post-closure care, is not demonstrated either to EPA or a State through the financial test or any other financial assurance mechanism specified in Rule II-808 and Rule I-806 equivalent or substantially equivalent State or Federal mechanisms. The current closure and/or post-closure cost estimates not covered by such financial assurance are shown for each facility:  
None.

This owner or operator is required to file a Form 10K with the Securities and Exchange Commission (SEC) for the latest fiscal year.

The fiscal year of this owner or operator ends on December 31. The figures for the following items marked with an asterisk are derived from this owner's or operator's independently audited, year-end financial statements for the latest completed fiscal year, ended December 31, 1985.

ALTERNATIVE I

1a. Sum of current closure and post-closure cost estimates (total of all cost estimates listed above) .....	\$ 1,370,300
1b. Sum of current closure and post-closure cost estimates for all facilities located in Puerto Rico.....	\$ 165,500
2. Amount of annual aggregate liability coverage to be demonstrated .....	\$ 2,000,000
3a. Sum of line 1(a) and 2 .....	\$ 3,370,300
3b. Sum of line 1(b) and 2 .....	\$ 2,165,500
*4. Total liabilities (if any portion of your closure or post- closure cost estimates is included in your total liabilities you can deduct that portion from this line and add that amount to lines 5 and 6) .....	\$1,566,000,000
*5. Tangible net worth .....	\$2,293,400,000
*6. Net worth .....	\$2,387,700,000
*7. Current assets .....	\$1,940,200,000
*8. Current liabilities .....	\$1,045,800,000
9. Net working capital (line 7 minus line 8) .....	\$ 894,400,000

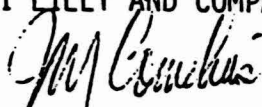
- \*10. The sum of net income plus depreciation, depletion, and amortization ..... \$ 653,800,000
- \*11. Total assets in U.S. (required only if less than 90% of assets are located in the U.S.) ..... \$3,090,700,000
- \*12. Total assets in Puerto Rico ..... more than \$ 100,000,000

	YES	NO
13. Is line 5 at least \$10 million ? .....	<u>X</u>	—
14. Is line 5 at least 6 times line 3(a)? .....	<u>X</u>	—
15. Is line 9 at least 6 times line 3(a)? .....	<u>X</u>	—
*16. Are at least 90% of assets located in the U.S.? If not, complete line 17 .....	—	<u>X</u>
17. Is line 11 at least 6 times line 3(a)? .....	<u>X</u>	—
18. Is line 12 at least 4 times line 3(b)? .....	<u>X</u>	—
19. Is line 4 divided by line 6 less than 2.0? .....	<u>X</u>	—
20. Is line 10 divided by line 4 greater than 0.1? .....	<u>X</u>	—
21. Is line 7 divided by line 8 greater than 1.5? .....	<u>X</u>	—

I hereby certify that the wording of this letter is identical to the wording specified in Rule II-808 J(7) as such regulations were constituted on the date shown immediately below.

Yours truly,

ELI LILLY AND COMPANY

  
J. M. Cornelius  
Vice President of Finance

March 31, 1986

pw

Attachments: Eli Lilly and Company - Annual Report 1985  
Statement of Ernst & Whinney - Auditors

bcc: Mrs. J. T. Hessler  
Mr. J. J. Rivera  
Mr. M. L. Esarey (3)

**CORPORATE GUARANTEE FOR CLOSURE OR  
POST-CLOSURE CARE OR LIABILITY COVERAGE**

Guarantee made this March 31, 1986, by Eli Lilly and Company, a business corporation organized under the laws of the State of Indiana, herein referred to as guarantor, to the EQB, obligee, on behalf of our subsidiary Eli Lilly Industries, Inc., of Road #2, KM 146.7, Mayaguez, Puerto Rico 00708.

**Recitals**

1. Guarantor meets or exceeds the financial test criteria and agrees to comply with the reporting requirement for guarantors as specified in Rules II-808 D(6), II-808 F(6), I-806 D(5), and I-806 F(5).

2. Eli Lilly Industries, Inc., owns or operates the following hazardous waste management facility covered by this guarantee: Eli Lilly Industries, Inc., of Road #2, KM 146.7, Mayaguez, Puerto Rico 00708 [PRD 0910247864], guarantee for closure and liability.

3. "Closure plans" and "post-closure plans" as used below refer to the plans maintained as required by Rules II-807 and I-805 for the closure and post-closure care of facilities as identified above.

4(a). For value received from Eli Lilly Industries, Inc., guarantor guarantees to EQB that in the event that Eli Lilly Industries, Inc. fails to perform closure of the above facility in accordance with the closure or post-closure plans and other permit or interim status requirements whenever required to do so, the guarantor shall do so or establish a trust fund as specified in Rules II-808 or I-806 as applicable, in the name of Eli Lilly Industries, Inc. in the amount of the current closure or post-closure cost estimates as specified in Rules II-808 and I-806.



4(b). For value received from Eli Lilly Industries, Inc., the guarantor guarantees to EQB that in the event that Eli Lilly Industries, Inc. fail to satisfy liability judgements arising from the operations of the above named facility, the guarantor agrees to satisfy such judgement(s), up to the limits required by these regulations for claims arising from the operations of the facilities from sudden and accidental occurrences that cause injuries to persons or property, or provide alternate liability coverage as specified in Rules I-806 and II-808 in the name of the owner or operator.

5. Guarantor agrees that if, at the end of any fiscal year before termination of this guarantee, the guarantor fails to meet the financial test criteria, guarantor shall send within 90 days, by certified mail, notice to the EQB for facilities located in Puerto Rico and to Eli Lilly Industries, Inc. that he intends to provide alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, in the name of Eli Lilly Industries, Inc. Within 120 days after the end of such fiscal year, the guarantor shall establish such financial assurance and/or liability coverage unless Eli Lilly Industries, Inc. has done so.

6. The guarantor agrees to notify the EQB by certified mail, of a voluntary or involuntary proceeding under Title 11 (Bankruptcy), U.S. Code, naming guarantor as debtor, within 10 days after commencement of the proceeding.

7. Guarantor agrees that within 30 days after being notified by the EQB of determination that guarantor no longer meets the financial test criteria or that he is disallowed from continuing as a guarantor of closure or post-closure care or liability coverage, he shall establish alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, in the name of Eli Lilly Industries, Inc., unless Eli Lilly Industries, Inc. has done so.

8(a). Guarantor agrees to remain bound under this guarantee notwithstanding any or all of the following: amendments or modification of the closure or post-closure plan, amendment or modification of the permit, the extension or reduction of the time of performance of closure or post-closure, or any other modification or alteration of an obligation of the owner or operator pursuant to Part VIII-I and II of this regulation.

8(b). Guarantor agrees to remain bound under this guarantee notwithstanding any or all of the following: amendment or modification of the liability requirements set by Rules II-808 H and I-806 H; variances establish according to the Rules II-808 H and I-806 H; or any other modification or alteration of an obligation of the owner or operator.

9. Guarantor agrees to remain bound under this guarantee for so long as Eli Lilly Industries, Inc. must comply with the applicable financial assurance or liability requirements of Rules II-808 and I-806 for the above-listed facilities, except that guarantor may cancel this guarantee by sending notice by certified mail to the EQB and to Eli Lilly Industries, Inc, such cancellation to become effective no earlier than 120 days after receipt of such notice by both the EQB and Eli Lilly Industries, Inc., as evidenced by the return receipts.

10. Guarantor agrees that if Eli Lilly Industries, Inc. fails to provide alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, and obtain written approval of such assurance from the EQB Chairman within 90 days after a notice of cancellation by the guarantor is received by the EQB from guarantor, guarantor shall provide such alternate financial assurance and/or liability coverage in the name of Eli Lilly Industries, Inc.

11. Guarantor expressly waives notice of acceptance of this guarantee by the EQB or by Eli Lilly Industries, Inc. Guarantor also expressly waives notice of amendments or modifications of the closure and/or post-closure plan and of amendments or modifications of the facility permit.


I hereby certify that the wording of this guarantee is identical to the wording specified in Rule II-808 J(8) as such regulations were constituted on the date first above written.

Effective date: March 31, 1986

ELI LILLY AND COMPANY



J. M. Cornelius  
Vice President of Finance

  
Signature of Witness or Notary

SUSAN K. WELSH

Resident of Marion County  
My Commission Expires  
January 27, 1989

# Ernst & Whinney

One Indiana Square, Suite 3400  
Indianapolis, Indiana 46204

317/236-1100

March 25, 1986

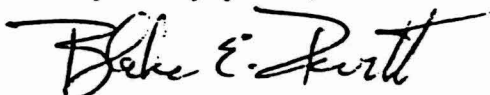
TO: Whom it May Concern

RE: Financial Data reported in letter of Mr. J.M. Cornelius dated  
March 31, 1986, addressed to the Environmental Quality Board

I have compared the financial data in items 4, 5, 6, 7, 8, 9, 10, 11, 12, and 16 listed on Pages 3 and 4 in the attached letter signed by Mr. Cornelius and dated March 31, 1986, with the audited consolidated financial statements of Eli Lilly and Company for the year ended December 31, 1985, upon which we have issued our report dated February 6, 1986.

The financial data referred to above is in agreement with the audited financial statements, and no matters came to my attention which cause me to believe that the data should be adjusted.

Very truly yours,



Blake E. Devitt  
Partner

BED:ngk  
Attachment



DEPARTMENT OF TRANSPORTATION  
UNITED STATES COAST GUARD

MAILING ADDRESS:

Supervisor, MSD Ponce  
P.O. Box 34, Playa Sta.  
Ponce, P.R. 00734-3034

16000  
30 June 1982

Mr. Vicente Diaz Diaz  
Eli Lilly Industries, Inc.  
P.O. Box 1748  
Mayaguez, PR 00709

Dear Mr. Diaz:

I would like to take this opportunity to thank you for the tour of your facilities in Mayaguez on 29 June, 1982. The tour of your plant identifying hazardous waste storage procedures and the varied products handled was very informative. I am also acknowledging receipt of your Hazardous Waste Management Plan, revision 6-2-82.

Should you have any questions concerning the Coast Guard or if I may assist you in any way please do not hesitate to contact me.

Sincerely,

*F. L. Whipple*  
F. L. WHIPPLE  
Lt. U.S. Coast Guard


RECEIVED  
1111 P.M.

23 de junio de 1982

Eli Lilly Industries, Inc.  
P.O. Box 1748  
Mayaguez, Puerto Rico 00709

Estimados Señores:

El día 23 de junio de 1982 visité las facilidades de Eli Lilly Industries, Inc., Mayaguez, Puerto Rico, en la cual el Ingeniero Vicente Díaz Díaz me mostró las diferentes áreas de su planta; así como conocer sus entradas y posibles rutas de evacuación, para poder familiarizarme con las mismas en caso de surgir alguna emergencia.

  
Juan A. Castillo  
Capitán de la Policía  
de Puerto Rico  
Area de Mayaguez

5-5320

## TRUST AGREEMENT

Trust Agreement, the "Agreement," entered into as of July 6, 1982, by and between Eli Lilly Industries, Inc., a Delaware corporation, the "Grantor," and The Indiana National Bank, a national bank, the "Trustee."

Whereas, the United States Environmental Protection Agency, "EPA," an agency of the United States Government, has established certain regulations applicable to the Grantor, requiring that an owner or operator of a hazardous waste management facility shall provide assurance that funds will be available when needed for closure and/or post-closure care of the facility,

Whereas, the Grantor has elected to establish a trust to provide all or part of such financial assurance for the facilities identified herein,

Whereas, the Grantor, acting through its duly authorized officers, has selected the Trustee to be the trustee under this agreement, and the Trustee is willing to act as trustee,

Now, Therefore, the Grantor and the Trustee agree as follows:

Section 1. Definitions. As used in this Agreement:

(a) The term "Grantor" means the owner or operator who enters into this Agreement and any successors or assigns of the Grantor.

(b) The term "Trustee" means the Trustee who enters into this Agreement and any successor Trustee.

Section 2. Identification of Facilities and Cost Estimates. This Agreement pertains to the facilities and cost estimates identified on attached Schedule A.

Section 3. Establishment of Fund. The Grantor and the Trustee hereby establish a trust fund, the "Fund," for the benefit of EPA. The Grantor and the Trustee intend that no third party have access to the Fund except as herein provided. The Fund is established initially as consisting of the property, which is acceptable to the Trustee, described in Schedule B attached hereto. Such property and any other property subsequently transferred to the Trustee is referred to as the Fund, together with all earnings and profits thereon, less any payments or distributions made by the Trustee pursuant to this Agreement. The Fund shall be held by the Trustee, IN TRUST, as hereinafter provided. The Trustee shall not be responsible nor shall it undertake any responsibility for the amount or adequacy of, nor any duty to collect from the Grantor, any payments necessary to discharge any liabilities of the Grantor established by EPA.

Section 4. Payment for Closure and Post-Closure Care. The Trustee shall make payments from the Fund as the EPA Regional Administrator

shall direct, in writing, to provide for the payment of the costs of closure and/or post-closure care of the facilities covered by this Agreement. The Trustee shall reimburse the Grantor or other persons as specified by the EPA Regional Administrator from the Fund for closure and post-closure expenditures in such amounts as the EPA Regional Administrator shall direct in writing. In addition, the Trustee shall refund to the Grantor such amounts as the EPA Regional Administrator specifies in writing. Upon refund, such funds shall no longer constitute part of the Fund as defined herein.

Section 5. Payments Comprising the Fund. Payments made to the Trustee for the Fund shall consist of cash or securities acceptable to the Trustee.

Section 6. Trustee Management. The Trustee shall invest and reinvest the principal and income of the Fund and keep the Fund invested as a single fund, without distinction between principal and income, in accordance with general investment policies and guidelines which the Grantor may communicate in writing to the Trustee from time to time, subject, however, to the provisions of this Section. In investing, reinvesting, exchanging, selling, and managing the Fund, the Trustee shall discharge his duties with respect to the trust fund solely in the interest of the beneficiary and with the care, skill, prudence, and diligence under the circumstances then prevailing which persons of prudence, acting in a like capacity and familiar with such matters, would use in the conduct of an enterprise of a like character and with like aims; except that:

(i) Securities or other obligations of the Grantor, or any other owner or operator of the facilities, or any of their affiliates as defined in the Investment Company Act of 1940, as amended, 15 U.S.C. 80a-2.(a), shall not be acquired or held, unless they are securities or other obligations of the Federal or a State government;

(ii) The Trustee is authorized to invest the Fund in time or demand deposits of the Trustee, to the extent insured by an agency of the Federal or State government; and

(iii) The Trustee is authorized to hold cash awaiting investment or distribution uninvested for a reasonable time and without liability for the payment of interest thereon.

Section 7. Commingling and Investment. The Trustee is expressly authorized in its discretion:

(a) To transfer from time to time any or all of the assets of the Fund to any common, commingled, or collective trust fund created by the Trustee in which the Fund is eligible to participate, subject to all of the provisions thereof, to be commingled with the assets of other trusts participating therein; and

(b) To purchase shares in any investment company registered under the Investment Company Act of 1940, 15 U.S.C. 80a-1 et seq., including one which may be created, managed, underwritten, or to which investment



advice is rendered or the shares of which are sold by the Trustee. The Trustee may vote such shares in its discretion.

**Section 8. Express Powers of Trustee.** Without in any way limiting the powers and discretions conferred upon the Trustee by the other provisions of this Agreement or by law, the Trustee is expressly authorized and empowered:

(a) To sell, exchange, convey, transfer, or otherwise dispose of any property held by it, by public or private sale. No person dealing with the Trustee shall be bound to see to the application of the purchase money or to inquire into the validity or expediency of any such sale or other disposition;

(b) To make, execute, acknowledge, and deliver any and all documents of transfer and conveyance and any and all other instruments that may be necessary or appropriate to carry out the powers herein granted;

(c) To register any securities held in the Fund in its own name or in the name of a nominee and to hold any security in bearer form or in book entry, or to combine certificates representing such securities with certificates of the same issue held by the Trustee in other fiduciary capacities, or to deposit or arrange for the deposit of such securities in a qualified central depository even though, when so deposited, such securities may be merged and held in bulk in the name of the nominee of such depository with other securities deposited therein by another person, or to deposit or arrange for the deposit of any securities issued by the United States Government, or any agency or instrumentality thereof, with a Federal Reserve bank, but the books and records of the Trustee shall at all times show that all such securities are part of the Fund;

(d) To deposit any cash in the Fund in interest-bearing accounts maintained or savings certificates issued by the Trustee, in its separate corporate capacity, or in any other banking institution affiliated with the Trustee, to the extent insured by an agency of the Federal or State government; and

(e) To compromise or otherwise adjust all claims in favor of or against the Fund.

**Section 9. Taxes and Expenses.** All taxes of any kind that may be assessed or levied against or in respect of the Fund and all brokerage commissions incurred by the Fund shall be paid from the Fund. All other expenses incurred by the Trustee in connection with the administration of this Trust, including fees for legal services rendered to the Trustee, the compensation of the Trustee to the extent not paid directly by the Grantor, and all other proper charges and disbursements of the Trustee shall be paid from the Fund.

**Section 10. Annual Valuation.** The Trustee shall annually, at least 30 days prior to the anniversary date of establishment of the Fund, furnish to the Grantor and to the appropriate EPA Regional Administrator a statement confirming the value of the Trust. Any securities

in the Fund shall be valued at market value as of no more than 60 days prior to the anniversary date of establishment of the Fund. The failure of the Grantor to object in writing to the Trustee within 90 days after the statement has been furnished to the Grantor and the EPA Regional Administrator shall constitute a conclusively binding assent by the Grantor, barring the Grantor from asserting any claim or liability against the Trustee with respect to matters disclosed in the statement.

Section 11. Advice of Counsel. The Trustee may from time to time consult with counsel, who may be counsel to the Grantor, with respect to any question arising as to the construction of this Agreement or any action to be taken hereunder. The Trustee shall be fully protected, to the extent permitted by law, in acting upon the advice of counsel.

Section 12. Trustee Compensation. The Trustee shall be entitled to reasonable compensation for its services as agreed upon in writing from time to time with the Grantor.

Section 13. Successor Trustee. The Trustee may resign or the Grantor may replace the Trustee, but such resignation or replacement shall not be effective until the Grantor has appointed a successor trustee and this successor accepts the appointment. The successor trustee shall have the same powers and duties as those conferred upon the Trustee hereunder. Upon the successor trustee's acceptance of the appointment, the Trustee shall assign, transfer, and pay over to the successor trustee the funds and properties then constituting the Fund. If for any reason the Grantor cannot or does not act in the event of the resignation of the Trustee, the Trustee may apply to a court of competent jurisdiction for the appointment of a successor trustee or for instructions. The successor trustee shall specify the date on which it assumes administration of the trust in a writing sent to the Grantor, the EPA Regional Administrator, and the present Trustee by certified mail 10 days before such change becomes effective. Any expenses incurred by the Trustee as a result of any of the acts contemplated by this Section shall be paid as provided in Section 9.

Section 14. Instructions to the Trustee. All orders, requests, and instructions by the Grantor to the Trustee shall be in writing, signed by such persons as are designated in the attached Exhibit A or such other designees as the Grantor may designate by amendment to Exhibit A. The Trustee shall be fully protected in acting without inquiry in accordance with the Grantor's orders, requests, and instructions. All orders, requests, and instructions by the EPA Regional Administrator to the Trustee shall be in writing, signed by the EPA Regional Administrators of the Regions in which the facilities are located, or their designees, and the Trustee shall act and shall be fully protected in acting in accordance with such orders, requests, and instructions. The Trustee shall have the right to assume, in the absence of written notice to the contrary, that no event constituting a change or a termination of the authority of any person to act on behalf of the Grantor or EPA hereunder has occurred. The Trustee shall have no duty to act in the absence of such orders, requests, and instructions from the Grantor and/or EPA, except as provided for herein.

**Section 15. Notice of Nonpayment.** The Trustee shall notify the Grantor and the appropriate EPA Regional Administrator, by certified mail within 10 days following the expiration of the 30-day period after the anniversary of the establishment of the Trust, if no payment is received from the Grantor during that period. After the pay-in period is completed, the Trustee shall not be required to send a notice of nonpayment.

**Section 16. Amendment of Agreement.** This Agreement may be amended by an instrument in writing executed by the Grantor, the Trustee, and the appropriate EPA Regional Administrator, or by the Trustee and the appropriate EPA Regional Administrator if the Grantor ceases to exist.

**Section 17. Irrevocability and Termination.** Subject to the right of the parties to amend this Agreement as provided in Section 16, this Trust shall be irrevocable and shall continue until terminated at the written agreement of the Grantor, the Trustee, and the EPA Regional Administrator, or by the Trustee and the EPA Regional Administrator, if the Grantor ceases to exist. Upon termination of the Trust, all remaining trust property, less final trust administration expenses, shall be delivered to the Grantor.

**Section 18. Immunity and Indemnification.** The Trustee shall not incur personal liability of any nature in connection with any act or omission, made in good faith, in the administration of this Trust, or in carrying out any directions by the Grantor or the EPA Regional Administrator issued in accordance with this Agreement. The Trustee shall be indemnified and saved harmless by the Grantor or from the Trust Fund, or both, from and against any personal liability to which the Trustee may be subjected by reason of any act or conduct in its official capacity, including all expenses reasonably incurred in its defense in the event the Grantor fails to provide such defense.

**Section 19. Choice of Law.** This Agreement shall be administered, construed, and enforced according to the laws of the State of Indiana.

**Section 20. Interpretation.** As used in this Agreement, words in the singular include the plural and words in the plural include the singular. The descriptive headings for each Section of this Agreement shall not affect the interpretation or the legal efficacy of this Agreement.

**Section 21. Exceptions to the Requirements of this Trust Agreement.** Until the standby trust fund is funded pursuant to the requirements of 40 Code of Federal Regulations § 265.143, the following are not required: (A) Payments into the trust fund; (B) Updating of Schedule A of this trust agreement to show current closure cost estimates; (C) Annual valuations as required by this trust agreement; and (D) Notices of nonpayment as required by this trust agreement.

In Witness Whereof the parties have caused this Agreement to be executed by their respective officers duly authorized and their corporate seals to be hereunto affixed and attested as of the date first above written: The parties below certify that the wording of this

Agreement is identical to the wording specified in 40 CFR 264.151(a)(1) as such regulations were constituted on the date first above written, except that Section 21 has been added to indicate those obligations of the Grantor or the Trustee in this Trust Agreement that are not applicable until the standby trust fund has been funded, based on 40 CFR § 265.143(b)(3)(ii)(A) - (D), as that regulation was constituted on the date first above written.

Eli Lilly Industries, Inc.

Attest:

J. Z. Z...  
Title: Treasurer

[Seal]

By [Signature]  
President  
Title

Commonwealth of Puerto Rico  
Affidavit no. 352



On this 23rd day of May, 1983, before me personally came J. M. Mann to me known, who, being by me duly sworn, did depose and say that he is the President of Eli Lilly Industries, Inc., the corporation described in and executed the above instrument; that he knows the seal of said corporation; that the seal affixed to such instrument is such corporate seal; that it was so affixed by order of the Board of Directors of said corporation, that he signed his name thereto by like order; at Carolina, Puerto Rico.

Notary Public

Attest:

[Signature]  
Title  
TRUST OFFICER

[Seal]

The Indiana National Bank

By [Signature]  
VICE PRESIDENT & TRUST OFFICER  
Title

**Schedule A**

**Eli Lilly Industries, Inc.  
Kilometer 146.7 State Road 2  
Mayaguez, Puerto Rico**

**EPA Identification Number: PRT 000010016**

**Closure Cost Estimates: \$92,000.00**

**Schedule B**

**Proceeds payable under the Seaboard Surety Company Financial Guarantee Bond, effective July 6, 1982, of which Eli Lilly Industries, Inc. is the Principal.**

A P P E N D I X    I I I

*Lilly*

**Eli Lilly and Company**

307 East McCarty Street  
Indianapolis, Indiana 46285  
(317) 261-2000

March 31, 1986

CERTIFIED MAIL  
RETURN RECEIPT REQUESTED

Environmental Quality Board  
P.O. Box 11488  
Santurce, Puerto Rico 00910-1488

Attention: Chairman, Environmental  
Quality Board

Dear Sir:

I am the chief financial officer of Eli Lilly and Company, Lilly Corporate Center, Indianapolis, Indiana 46285. This letter is in support of the use of the financial test to demonstrate financial responsibility for liability coverage and closure and/or post-closure care, as specified in Rules I-806 and II-808.

The owner or operator identified above is the owner or operator of the following facilities for which liability coverage is being demonstrated through the financial test and/or corporate guarantee specified in Rules I-806 and II-808.

<u>Identification No.</u>	<u>Name/Address</u>	
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	Financial Test
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	Financial Test
IND 006421325	Eli Lilly and Company Materials Center 1402 South Dakota Street Indianapolis, Indiana 46285	Financial Test



*Lilly*

**Eli Lilly and Company**

307 East McCarty Street  
Indianapolis, Indiana 46285  
(317) 261-2000

March 31, 1986

CERTIFIED MAIL  
RETURN RECEIPT REQUESTED

Environmental Quality Board  
P.O. Box 11488  
Santurce, Puerto Rico 00910-1488

Attention: Chairman, Environmental  
Quality Board

Dear Sir:

I am the chief financial officer of Eli Lilly and Company, Lilly Corporate Center, Indianapolis, Indiana 46285. This letter is in support of the use of the financial test to demonstrate financial responsibility for liability coverage and closure and/or post-closure care, as specified in Rules I-806 and II-808.

The owner or operator identified above is the owner or operator of the following facilities for which liability coverage is being demonstrated through the financial test and/or corporate guarantee specified in Rules I-806 and II-808.

<u>Identification No.</u>	<u>Name/Address</u>	
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	Financial Test
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	Financial Test
IND 006421325	Eli Lilly and Company Materials Center 1402 South Dakota Street Indianapolis, Indiana 46285	Financial Test



PRD 091024786

Eli Lilly Industries, Inc      Corporate.  
Mayaguez Plant                      Guarantee  
Road #2, KM 146.7  
Mayaguez, Puerto Rico 00708

1. The owner or operator identified above owns or operates the following facilities for which financial assurance for closure or post-closure care is demonstrated through the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by the test are shown for each facility:

<u>Iden. No.</u>	<u>Name / Address</u>	<u>Closure Cost (\$)</u>
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	\$562,000
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	569,000
IND 006421325	Eli Lilly and Company Materials Center 1402 S. Dakota Street Indianapolis, Indiana 46285	73,800

2. The owner or operator identified above guarantees, through the corporate guarantee specified in Rules I-806 and II-808, the closure and post-closure care of the following facilities owned or operated by its subsidiaries. The current cost estimates for the closure or post-closure care so guaranteed are shown for each facility:

PRD 091024786	Eli Lilly Industries, Inc. Mayaguez Plant Road #2, KM 146.7 Mayaguez, Puerto Rico 00708	\$165,500
---------------	--	-----------

3. Where the Commonwealth of Puerto Rico is not administering the financial requirements of Rules I-806 and II-808, this owner or operator is demonstrating financial assurance for the closure or post-closure care of the following facilities through the use of a test equivalent or substantially equivalent to the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by such a test are shown for each facility:  
NONE

PRD 091024786

Eli Lilly Industries, Inc  
Mayaguez Plant  
Road #2, KM 146.7  
Mayaguez, Puerto Rico 00708

Corporate.  
Guarantee

1. The owner or operator identified above owns or operates the following facilities for which financial assurance for closure or post-closure care is demonstrated through the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by the test are shown for each facility:

<u>Iden. No.</u>	<u>Name / Address</u>	<u>Closure Cost (\$)</u>
IND 006050967	Eli Lilly and Company Tippecanoe Laboratories Lilly Road Lafayette, Indiana 47902	\$562,000
IND 072040348	Eli Lilly and Company Clinton Laboratories Clinton, Indiana 47842	569,000
IND 006421325	Eli Lilly and Company Materials Center 1402 S. Dakota Street Indianapolis, Indiana 46285	73,800

2. The owner or operator identified above guarantees, through the corporate guarantee specified in Rules I-806 and II-808, the closure and post-closure care of the following facilities owned or operated by its subsidiaries. The current cost estimates for the closure or post-closure care so guaranteed are shown for each facility:

PRD 091024786	Eli Lilly Industries, Inc. Mayaguez Plant Road #2, KM 146.7 Mayaguez, Puerto Rico 00708	\$165,500
---------------	--	-----------

3. Where the Commonwealth of Puerto Rico is not administering the financial requirements of Rules I-806 and II-808, this owner or operator is demonstrating financial assurance for the closure or post-closure care of the following facilities through the use of a test equivalent or substantially equivalent to the financial test specified in Rules I-806 and II-808. The current closure and/or post-closure cost estimates covered by such a test are shown for each facility:  
NONE

4. The owner or operator identified above owns or operates the following hazardous waste management facilities for which financial assurance for closure or, if a disposal facility, post-closure care, is not demonstrated either to EPA or a State through the financial test or any other financial assurance mechanism specified in Rule II-808 and Rule I-806 equivalent or substantially equivalent State or Federal mechanisms. The current closure and/or post-closure cost estimates not covered by such financial assurance are shown for each facility:  
None.

This owner or operator is required to file a Form 10K with the Securities and Exchange Commission (SEC) for the latest fiscal year.

The fiscal year of this owner or operator ends on December 31. The figures for the following items marked with an asterisk are derived from this owner's or operator's independently audited, year-end financial statements for the latest completed fiscal year, ended December 31, 1985.

ALTERNATIVE I

1a. Sum of current closure and post-closure cost estimates (total of all cost estimates listed above) .....	\$ 1,370,300
1b. Sum of current closure and post-closure cost estimates for all facilities located in Puerto Rico.....	\$ 165,500
2. Amount of annual aggregate liability coverage to be demonstrated .....	\$ 2,000,000
3a. Sum of line 1(a) and 2 .....	\$ 3,370,300
3b. Sum of line 1(b) and 2 .....	\$ 2,165,500
*4. Total liabilities (if any portion of your closure or post- closure cost estimates is included in your total liabilities you can deduct that portion from this line and add that amount to lines 5 and 6) .....	\$1,566,000,000
*5. Tangible net worth .....	\$2,293,400,000
*6. Net worth .....	\$2,387,700,000
*7. Current assets .....	\$1,940,200,000
*8. Current liabilities .....	\$1,045,800,000
9. Net working capital (line 7 minus line 8) .....	\$ 894,400,000

- \*10. The sum of net income plus depreciation, depletion,  
and amortization ..... \$ 653,800,000
- \*11. Total assets in U.S. (required only if less than  
90% of assets are located in the U.S.) ..... \$3,090,700,000
- \*12. Total assets in Puerto Rico ..... more than \$ 100,000,000

	YES	NO
13. Is line 5 at least \$10 million ? .....	<u>X</u>	___
14. Is line 5 at least 6 times line 3(a)? .....	<u>X</u>	___
15. Is line 9 at least 6 times line 3(a)? .....	<u>X</u>	___
*16. Are at least 90% of assets located in the U.S.? If not, complete line 17 .....	___	<u>X</u>
17. Is line 11 at least 6 times line 3(a)? .....	<u>X</u>	___
18. Is line 12 at least 4 times line 3(b)? .....	<u>X</u>	___
19. Is line 4 divided by line 6 less than 2.0? .....	<u>X</u>	___
20. Is line 10 divided by line 4 greater than 0.1? .....	<u>X</u>	___
21. Is line 7 divided by line 8 greater than 1.5? .....	<u>X</u>	___

I hereby certify that the wording of this letter is identical to the  
wording specified in Rule II-808 J(7) as such regulations were constituted  
on the date shown immediately below.

Yours truly,

ELI LILLY AND COMPANY

  
J. M. Cornelius  
Vice President of Finance

March 31, 1986

pw

Attachments: Eli Lilly and Company - Annual Report 1985

Statement of Ernst & Whinney - Auditors

bcc: Mrs. J. T. Hessler

Mr. J. J. Rivera

Mr. M. L. Esarey (3)

CORPORATE GUARANTEE FOR CLOSURE OR  
POST-CLOSURE CARE OR LIABILITY COVERAGE

Guarantee made this March 31, 1986, by Eli Lilly and Company, a business corporation organized under the laws of the State of Indiana, herein referred to as guarantor, to the EQB, obligee, on behalf of our subsidiary Eli Lilly Industries, Inc., of Road #2, KM 146.7, Mayaguez, Puerto Rico 00708.

Recitals

1. Guarantor meets or exceeds the financial test criteria and agrees to comply with the reporting requirement for guarantors as specified in Rules II-808 D(6), II-808 F(6), I-806 D(5), and I-806 F(5).

2. Eli Lilly Industries, Inc., owns or operates the following hazardous waste management facility covered by this guarantee: Eli Lilly Industries, Inc., of Road #2, KM 146.7, Mayaguez, Puerto Rico 00708 [PRD 0910247864], guarantee for closure and liability.

3. "Closure plans" and "post-closure plans" as used below refer to the plans maintained as required by Rules II-807 and I-805 for the closure and post-closure care of facilities as identified above.

4(a). For value received from Eli Lilly Industries, Inc., guarantor guarantees to EQB that in the event that Eli Lilly Industries, Inc. fails to perform closure of the above facility in accordance with the closure or post-closure plans and other permit or interim status requirements whenever required to do so, the guarantor shall do so or establish a trust fund as specified in Rules II-808 or I-806 as applicable, in the name of Eli Lilly Industries, Inc. in the amount of the current closure or post-closure cost estimates as specified in Rules II-808 and I-806.

4(b). For value received from Eli Lilly Industries, Inc., the guarantor guarantees to EQB that in the event that Eli Lilly Industries, Inc. fail to satisfy liability judgements arising from the operations of the above named facility, the guarantor agrees to satisfy such judgement(s), up to the limits required by these regulations for claims arising from the operations of the facilities from sudden and accidental occurrences that cause injuries to persons or property, or provide alternate liability coverage as specified in Rules I-806 and II-808 in the name of the owner or operator.

5. Guarantor agrees that if, at the end of any fiscal year before termination of this guarantee, the guarantor fails to meet the financial test criteria, guarantor shall send within 90 days, by certified mail, notice to the EQB for facilities located in Puerto Rico and to Eli Lilly Industries, Inc. that he intends to provide alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, in the name of Eli Lilly Industries, Inc. Within 120 days after the end of such fiscal year, the guarantor shall establish such financial assurance and/or liability coverage unless Eli Lilly Industries, Inc. has done so.

6. The guarantor agrees to notify the EQB by certified mail, of a voluntary or involuntary proceeding under Title 11 (Bankruptcy), U.S. Code, naming guarantor as debtor, within 10 days after commencement of the proceeding.

7. Guarantor agrees that within 30 days after being notified by the EQB of determination that guarantor no longer meets the financial test criteria or that he is disallowed from continuing as a guarantor of closure or post-closure care or liability coverage, he shall establish alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, in the name of Eli Lilly Industries, Inc., unless Eli Lilly Industries, Inc. has done so.

8(a). Guarantor agrees to remain bound under this guarantee notwithstanding any or all of the following: amendments or modification of the closure or post-closure plan, amendment or modification of the permit, the extension or reduction of the time of performance of closure or post-closure, or any other modification or alteration of an obligation of the owner or operator pursuant to Part VIII-I and II of this regulation.

8(b). Guarantor agrees to remain bound under this guarantee notwithstanding any or all of the following: amendment or modification of the liability requirements set by Rules II-808 H and I-806 H; variances establish according to the Rules II-808 H and I-806 H; or any other modification or alteration of an obligation of the owner or operator.

9. Guarantor agrees to remain bound under this guarantee for so long as Eli Lilly Industries, Inc. must comply with the applicable financial assurance or liability requirements of Rules II-808 and I-806 for the above-listed facilities, except that guarantor may cancel this guarantee by sending notice by certified mail to the EQB and to Eli Lilly Industries, Inc., such cancellation to become effective no earlier than 120 days after receipt of such notice by both the EQB and Eli Lilly Industries, Inc., as evidenced by the return receipts.

10. Guarantor agrees that if Eli Lilly Industries, Inc. fails to provide alternate financial assurance and/or liability coverage as specified in Rules II-808 or I-806, as applicable, and obtain written approval of such assurance from the EQB Chairman within 90 days after a notice of cancellation by the guarantor is received by the EQB from guarantor, guarantor shall provide such alternate financial assurance and/or liability coverage in the name of Eli Lilly Industries, Inc.



11. Guarantor expressly waives notice of acceptance of this guarantee by the EQB or by Eli Lilly Industries, Inc. Guarantor also expressly waives notice of amendments or modifications of the closure and/or post-closure plan and of amendments or modifications of the facility permit.

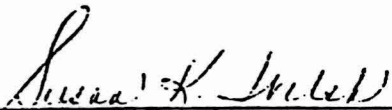
I hereby certify that the wording of this guarantee is identical to the wording specified in Rule II-808 J(8) as such regulations were constituted on the date first above written.

Effective date: March 31, 1986

ELI LILLY AND COMPANY



J. M. Cornelius  
Vice President of Finance



Signature of Witness or Notary

SUSAN K. WELSH

Resident of Marion County  
My Commission Expires  
January 27, 1989



Ernst & Whinney.

One Indiana Square, Suite 3400  
Indianapolis, Indiana 46204

317/236-1100

March 25, 1986


TO: Whom it May Concern

RE: Financial Data reported in letter of Mr. J.M. Cornelius dated  
March 31, 1986, addressed to the Environmental Quality Board

I have compared the financial data in items 4, 5, 6, 7, 8, 9, 10, 11, 12, and 16 listed on Pages 3 and 4 in the attached letter signed by Mr. Cornelius and dated March 31, 1986, with the audited consolidated financial statements of Eli Lilly and Company for the year ended December 31, 1985, upon which we have issued our report dated February 6, 1986.

The financial data referred to above is in agreement with the audited financial statements, and no matters came to my attention which cause me to believe that the data should be adjusted.

Very truly yours,



Blake E. Devitt  
Partner

BED:ngk  
Attachment

A P P E N D I X I V

ELI LILLY INDUSTRIES, INC.

M. 12.6 65TH. INFANTRY AVE.  
CAROLINA, PUERTO RICO  
P.O. BOX 71325  
SAN JUAN, PUERTO RICO 00936

KM. 146.7 STATE ROAD NO. 2  
MAYAGUEZ, PUERTO RICO  
P.O. BOX 1748  
MAYAGUEZ, PUERTO RICO 00709

June 4, 1982

CERTIFIED - RETURN RECEIPT REQUESTED

Mr. Ramón Valentín Cruz  
Regional Director  
Civil Defense  
Box 1042  
Mayaguez, Puerto Rico 00709

Dear Mr. Valentín:

On November 19, 1980, in accordance with the Resource Conservation and Recovery Act (RCRA) of 1976, the Environmental Protection Agency (EPA) in a cooperative arrangement with the Environmental Quality Board (EQB) of Puerto Rico, was directed to promulgate regulations to protect human health and the environment from the improper management of hazardous waste.

One of the requirements established on EPA, 40 CFR, Part 256.37, and EQB, Rule 810, is to familiarize the Civil Defense with the layout of the facilities, with the properties of hazardous waste handled at the plant, places where personnel would normally be working, entrances to roads inside the facilities and possible evacuation routes.

For this purpose we are inviting you, or your representatives, to visit our facilities, located at State Road No. 2, Km. 146.7, Mayagüez, Puerto Rico, at your earliest convenience.

If you have any questions, or if further information is needed, please do not hesitate and let me know.

Sincerely,

ELI LILLY INDUSTRIES, INC.



Vicente Díaz Díaz  
Dept. Head Engineering

VDD/yi

# ELI LILLY INDUSTRIES, INC.

KM. 12.6 65TH INFANTRY AVE.  
CAROLINA, PUERTO RICO

P.O. BOX 71325  
SAN JUAN, PUERTO RICO 00936

KM. 146.7 STATE ROAD NO. 2  
MAYAGUEZ, PUERTO RICO

P.O. BOX 1748  
MAYAGUEZ, PUERTO RICO 00708

June 4, 1982

**CERTIFIED - RETURN RECEIPT REQUESTED**

Mr. Israel Pabón, Chief  
Fire Department  
50 Nenadich St.  
Mayaguez, P. R. 00708

Dear Mr. Pabón:

On November 19, 1980, in accordance with the Resource Conservation and Recovery Act (RCRA) of 1976, the Environmental Protection Agency (EPA) in a cooperative arrangement with the Environmental Quality Board (EQB) of Puerto Rico, was directed to promulgate regulations to protect human health and the environment from the improper management of hazardous waste.

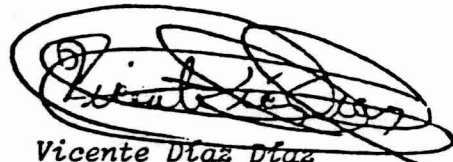
One of the requirements established on E.P.A., 40 CFR, Part 256.37 and E.Q.B., Rule 810, is to familiarize the Fire Department with the layout of the facilities, with the properties of hazardous waste handled at the plant, places where personnel would normally be working, entrances to roads inside the facilities and possible evacuation routes.

For this purpose we are inviting you, or your representatives, to visit our facilities, located at State Road No. 2, Km. 146.7, Mayaguez, Puerto Rico, at your earliest convenience.

If you have any questions, or if further information is needed, please do not hesitate and let me know.

Sincerely,

ELI LILLY INDUSTRIES, INC.



Vicente Diaz Diaz  
Dept. Head Engineering

VDD/yi

# ELI LILLY INDUSTRIES, INC.

KM. 12.6 65TH INFANTRY AVE.  
CAROLINA, PUERTO RICO

G.P.O. BOX 71325  
SAN JUAN, PUERTO RICO 00936

KM. 146.7 STATE ROAD NO. 2  
MAYAGÜEZ, PUERTO RICO

P.O. BOX 1748  
MAYAGÜEZ, PUERTO RICO 00709

June 4, 1982

CERTIFIED - RETURN RECEIPT REQUESTED

Mr. Julises Beauchamp, Lieutenant  
P.R. Police Department  
Box 461  
Añasco, P. R. 00610

Dear Mr. Beauchamp:

On November 19, 1980, in accordance with the Resource Conservation and Recovery Act (RCRA) of 1976, the Environmental Protection Agency (EPA) in a cooperative arrangement with the Environmental Quality Board (EQB) of Puerto Rico, was directed to promulgate regulations to protect human health and the environment from the improper management of hazardous waste.

One of the requirements established on E.P.A., 40 CFR, Part 256.37, and E.Q.B., Rule 810, is to familiarize the Police Department with the layout of the facilities, with the properties of hazardous waste handled at the plant, places where personnel would normally be working, entrances to roads inside the facilities and possible evacuation routes.

For this purpose we are inviting you, or your representatives, to visit our facilities, located at State Road No. 2, Km. 146.7, Mayagüez, Puerto Rico, at your earliest convenience.

If you have any questions, or if further information is needed, please do not hesitate and let me know.

Sincerely,

ELI LILLY INDUSTRIES, INC.



Vicente Diaz Blas  
Dept. Head Engineering

VDD/yi

ELI LILLY INDUSTRIES, INC.

KM. 12.6 65TH. INFANTRY AVE.  
CAROLINA, PUERTO RICO  
P.O. BOX 71325  
SAN JUAN, PUERTO RICO 00936

KM. 146.7 STATE ROAD NO. 2  
MAYAGUEZ, PUERTO RICO  
P.O. BOX 1746  
MAYAGUEZ, PUERTO RICO 00705

June 4, 1982

CERTIFIED - RETURN RECEIPT REQUESTED

Commander  
Coast Guard Department  
Greater Antilles Section  
Box 2029  
San Juan, Puerto Rico 00903

Attention: Captain Veran

Gentlemen:

On November 19, 1980, in accordance with the Resource Conservation and Recovery Act (RCRA) of 1976, the Environmental Protection Agency (EPA) in a cooperative arrangement with the Environmental Quality Board (EQB) of Puerto Rico, was directed to promulgate regulations to protect human health and the environment from the improper management of hazardous waste.

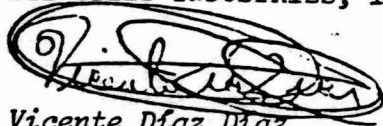
One of the requirements established on EPA, 40 CFR, Part 256.37, and EQB, Rule 810, is to familiarize the Coast Guard Department with the layout of the facilities, with the properties of hazardous waste handled at the plant, places where personnel would normally be working, entrances to roads inside the facilities and possible evacuation routes.

For this purpose we are inviting you, or your representatives, to visit our facilities, located at State Road No. 2, Km. 146.7, Mayagüez, Puerto Rico, at your earliest convenience.

If you have any questions, or if further information is needed, please do not hesitate and let me know.

Sincerely,

ELI LILLY INDUSTRIES, INC.



Vicente Diaz Diaz  
Dept. Head Engineering

VDD/yi

ESTADO LIBRE ASOCIADO DE PUERTO RICO  
POLICIA DE PUERTO RICO

*212*  
*Acordados*  
*Wagbe* *ell*

A/Ma/14/1/# 271

Distrito de Añasco, P.R.  
14 de junio de 1982.

MEMORANDO

A

Comandante Area Mayaguez  
Mayaguez, Puerto Rico

DE

*7-1-1982*  
*Inte. Ulicós Benachamp 7-5167*  
Comandante de Distrito

ASUNTO

EA-VIC CARTA

Adjunto carta del Sr. Vicente  
Díaz Díaz de la Fábrica Eli Lilly Industries, Inc.,  
por pertenecer la misma en el Distrito de Mayaguez.

RECEIVED

JUN 17 A.M.

CC :- Eli Lilly Industries Inc.  
P.O. Box 1748  
Mayaguez, Puerto Rico

A P P E N D I X V



## QUALITY ASSURANCE/QUALITY CONTROL

### CONTAINMENT SYSTEM

The sampling methods used to obtain representative samples of primary and secondary wastes are based on the containment system for the waste. Representative samples will be collected from tanks, containers (drums and palletainers) and tankers as follows:

1. Tanks - Once the primary or secondary tank is filled (90% of its total capacity) the waste will be agitated for 30 minutes to produce an homogeneous mixture. Then a sample will be drawn from the bottom of the tank. Every two weeks all samples from either system tank taken in this period of time will be combined to produce a composite sample. This will be the sample analyzed.
2. Containers (Drums and Palletainers) - Samples from each different wastes coming from Carolina in drums will be taken as specified in the following table:

<u>No. of Containers</u>	<u>No. of Containers Sampled</u>
2 - 15	2
16 - 40	3
41 - 65	5
66 - 110	7
111 - 180	10

This table is taken from the procedures recommended in the Military Standard 105 D. Each sample taken must be analyzed individually. Any replicates (tests) from each sample will be done following guidelines specified in the individual procedure for each test to be performed (as needed).

If one of the field replicates fails to meet characterization, material will be resampled and re-analyzed. If the results of replicates pass the test, the material will be accepted. If any of the resamples taken confirms failure, the material will be referred to the originator and it will be kept in hold until the originator confirms the information and makes corrective actions. Evidence that proves that the material has been correctly identified will be submitted and documented.

3. Tankers - A sample will be collected at the time it arrives at the plant.

Liquid - Wastes are sampled from the transporting container prior to unloading.

### Sampling Equipment and Methods

A Coliwassa, from Nasco Company shall be used to sample containers and tank trucks. The sampling procedure follows:

1. Clean the Coliwassa. Adjust the sampler's locking mechanism to assure that the stopper provides a tight closure. Open sampler by placing the stopper rod handled in the "T" position and pushing the rod down until the handle sits against the samplers locking block.
2. Slowly lower the sampler into the waste at a rate that permits the level of liquid inside and outside the sampler to remain the same. If the level of waste in the sampler tube is lower inside than it is outside, the sampling rate is too fast and will produce a non-representative sample.
3. When the sampler hits the bottom of the waste container, push the sampler tube down to close and lock the stopper by turning the "T" handle until it is upright and the end rests on the locking block.
4. Withdraw the Coliwassa from the waste and wipe the outside with a disposable cloth. Clean the Coliwassa.

The methods used for Quality Assurance and Quality Control are as follows:

1. Chain of Custody Procedures
  - a) Samples to be assayed in an off site laboratory.
  - b) Samples will be taken in DP 750 plastic bottles, or equivalent.
  - c) Bottles will be tightly closed and sealed with teflon or gummed paper seals.
  - d) Identification of samples will include the number of sample, name of collector, name of transporter, date and time of collection.

#### 2. Samples Control

For all samples collected from tanks, containers and tankers a field log book will be provided to record each sample. The log book will include number of sample, sample source, purpose of sample, type of waste, volume of sample taken, date and time of collection.

### 3. Instrument Decontamination

For instrument decontamination the methods used will be those recommended in the second edition (1982) of SW-846 under each test method.

### 4. Sample Preparation Method

The methods that will be used for sample preparation are those recommended in the second edition (1982) of SW-846 under each test method.

## QUALITY ASSURANCE/QUALITY CONTROL PROGRAM FOR TRIAL BURN

### A) Quality Assurance Organization and Responsibilities

The sample will be taken by a trained Environmental Control Senior Operator following sampling guidelines (enclosed in sampling information) and using adequate equipment (see sampling equipment). Once the appropriate samples are collected, it will be brought to the laboratory area and/or sent to an outside contracted private laboratory (following the appropriate chain of custody procedure).

Samples that are to be analyzed in our laboratory, are received and logged in the assigned log book (as stated in the chain of custody procedure) by a certified analyst. A certified and trained analyst will perform required tests following the approved methods.

The person in charge of the Environmental Control area will make regular inspections to records and analysis information (data) to assure that all requirements are being met. This person is not directly involved in the sampling or the analysis and is assigned the responsibility of ensuring that QC/QA measures are properly employed.

### B) Laboratory Performance Evaluation

An external laboratory will be used to evaluate and validate the Lilly Laboratory performance. The frequency of analysis made by the external laboratory will be determined for each parameter assayed.

C) Blanks

As a Quality Control Policy, distilled water blanks are to be analyzed to ensure all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank will be processed as a safeguard against laboratory contamination. Blank samples are to be carried through all stages of the sample preparation and measurement.

D) Procedures to asses precision of data

Field replicates are collected (as stated in the sampling procedure) to validate the precision of the analysis and ensure homogeneity of the sampling technique. Spikes are to be made to check recovery and accuracy as specified in methods procedures. In the case methods submitted do not include the spiking samples it will be added to the procedure and the percent of recovery will be calculated.

E) Data Validation

All the data related to spikes and duplicates (precision and accuracy studies) is to be recorded in a validation record book (this will include procedures and statistical information). Statistical parameters will be run to check minimum replicates required for each applicable test, and the replicates will be done as required in the validations.

F) Calibration Process

Laboratory equipment calibrations are to be performed as required under each individual procedure and/or following manufacturer's recommendations:

For G.C. tests calibration of the chromatographic system will be done using either the external standard or the internal standard technique. The system will be checked against secondary standards, to verify detection limits and sensitivity of the instrument.

G) Sample Containers, Preservation and Holding Times

Samples will be collected in glass vials (40 ml capacity screw cap vial: Pierce #13075 or equivalent). System will be teflon faced silicone (Pierce #12722 or equivalent). The bottles are filled in such a manner, that no air bubbles pass through the sample as the bottle is being filled. The bottle is sealed so that no air bubbles are entrapped in it.

No solid material is to interfere with the sealing of the glass vial. The hermetic seal on the sample bottle is to be maintained until time of analysis. The sample will be transferred to the analysis glassware by means of a hypodermic syringe. If the samples are not analyzed immediately, they must be refrigerated from the time of collection until analysis begins. If the sample contains free or combined chlorine, 10 mg/40 ml of sodium thiosulfate as preservative should be added to the empty sample vial just prior to sampling, shaking vigorously after collection. All preserved samples are to be analyzed within 14 days of collection.

H) Preventive Maintenance

Preventive Maintenance of laboratory equipment and instruments is given by using a computerized schedule system implemented by the Engineering department. The schedule follows the manufacturer's recommendation for each equipment and instrument.

TABLE I  
LIQUID WASTE ANALYSIS TEST METHODS

<u>PARAMETERS</u>	<u>METHOD DESCRIPTION</u>
Chemical Composition (quantification of compounds greater than or equal to 0.0 1%)	Determination of volatile organic compounds will be achieved following Method 8015 in SW846, involving direct injection of the sample into a gas chromatograph.
Chemical Composition	Determination of suspected Appendix VIII Compounds using Method 8030 (for ACN) and Method 8020 (for Toluot) in SW846, involving direct injection of sample into gas chromatograph.
Total Organic Chloride	Determination of chlorinated organic compounds using the 6C/MS Method 8240 in SW846.
Water Content	Determination of water content by Karl Fisher titration as per ASTM E 203.
pH	Determine the pH of the secondary waste using a pH meter with a colomel combination electrode as per Method 9040 in SW846.
Heat of Combustion	Determine the heat of combustion of the primary waste using an oxygen bomb calorimeter as per ASTM D240.
Specific Gravity	Determine the specific gravity using a digital density instrument or equivalent as per ASTM D891.
Ash Content	Determine the ash content of the wastes as per ASTM 482 using a nuttle furnace.
Viscosity	Determine the viscosity of the sample using a calibrated kinematic viscosimeter or equivalent as per ASTM D445.

APPENDIX VI INSPECTION LOG SAMPLES

CONTAINMENT SYSTEM INSPECTION LOG

ITEMS	GENERAL CONDITION	COMMENTS	ACTION TAKEN
1. Drums' Conditions a) leaks b) corrosion c) markings in floor d) labels e) pallets			
2. Drum Dike Conditions a) coating condition b) cracks on floors c) drain valve d) roof conditions f) signs conditions			
3. Operation Conditions a) entrance protection b) water in system c) no drums d) % capacity occupied			

INSPECTOR'S NAME: \_\_\_\_\_

SUPERVISOR : \_\_\_\_\_

DATE : \_\_\_\_\_

TIME : \_\_\_\_\_



**ELI LILLY INDUSTRIES, INC.  
MAYAGUEZ PLANT**

**HAZARDOUS WASTE STORAGE TANKS INSPECTION LOG**

TANK NO	WASTE STORED	GEN. TANK CONDITION	VALVE & PUMP CONDITION	DIKE CONDITION	COMMENTS/ ACTION TAKEN
	SECONDARY HAZARDOUS				
12	PRIMARY HAZARDOUS				
13A	PRIMARY HAZARDOUS				
14	SECONDARY HAZARDOUS				
14A	SECONDARY HAZARDOUS				
*	ACETONE SPENT				
*	ACETONE SPENT				
*	ETHYL ETHER SPENT				
*	ETHYL ACETATE SPENT				
*	TOLUENE SPENT				
34	ALCOHOL SPENT				
41	ACN SPENT				
42	ACN SPENT				

1. FOLLOWING ITEMS HAVE BEEN CHECKED IN EACH TANK:  
LEAKS, CORROSION, CRACKS, ELECTRICAL DEVICES CONDITION, DRAIN VALVE POSITION  
DIKE, NOTICE OF ANY SPILL, ETC.

TANKS WITH ASTERISKS STORES SPENT SOLVENTS FOR LESS THAN 90 DAYS. THEY HAVE BEEN CLASSIFIED AS HAZARDOUS BY DEFINITION.

SIGNATURE \_\_\_\_\_ DATE \_\_\_\_\_ TIME \_\_\_\_\_

## INCINERATOR DAILY INSPECTION LOG

MONTH \_\_\_\_\_

[illegible]

COMMENTS : \_\_\_\_\_